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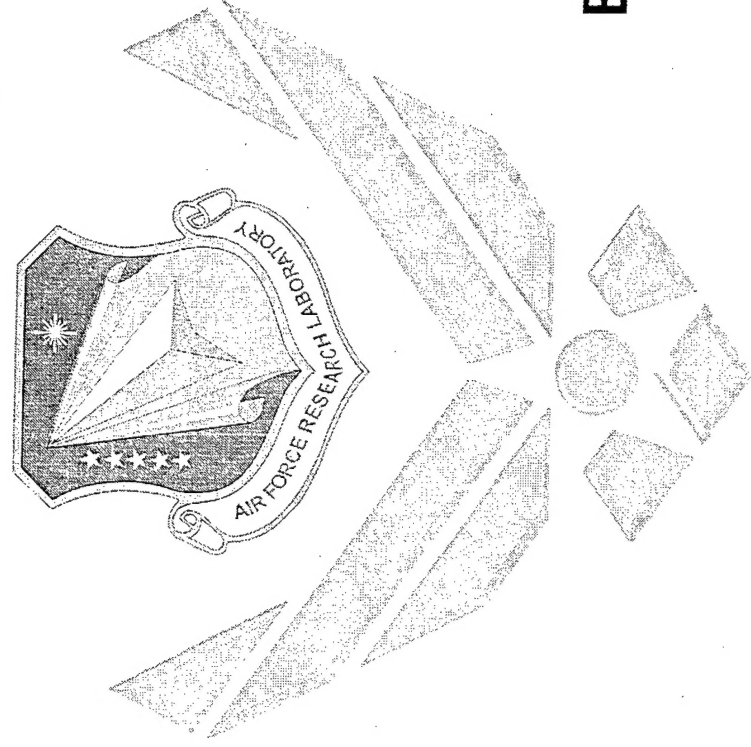
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				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S) J. M. Mabry, A. Vij, D. Marchant, B. D. Viers, P. N. Ruth, C. E. Schlaefer				5d. PROJECT NUMBER 2303	
				5e. TASK NUMBER M1A3	
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Fluorinated Polyhedral Oligomeric Silsesquioxanes (FluoroPOSS)

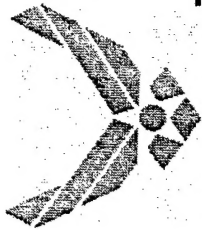


*Silicones and Silicone-
Modified Materials*

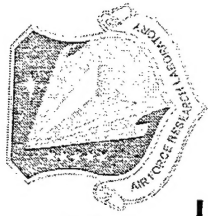
April 1, 2004

JM Mabry, A Vij, D Marchant,
BD Viers, PN Ruth, and CE Schlaefer
Polymer Working Group
Air Force Research Laboratory
(661)275-5857

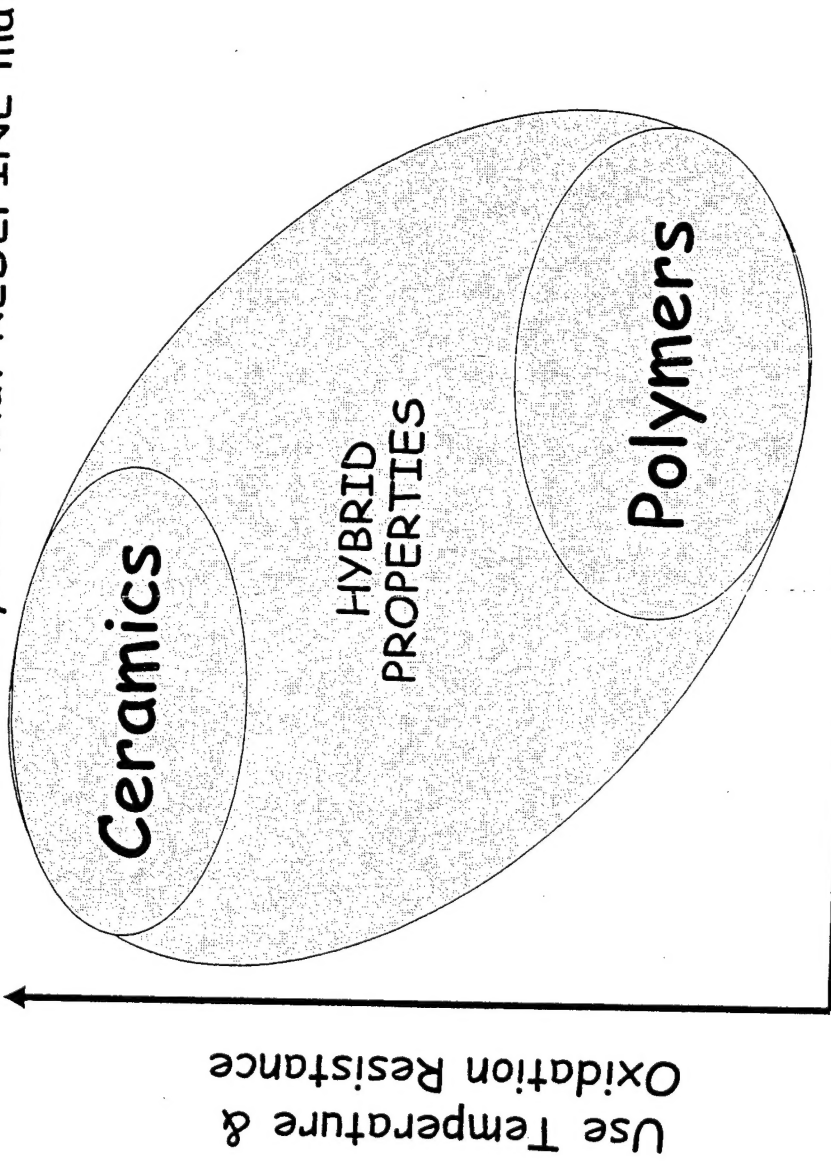
joseph.mabry@edwards.af.mil



Hybrid Inorganic/Organic Polymers



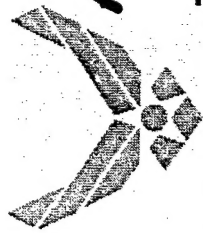
Goal: Develop High Performance Polymers that REDEFINE material properties



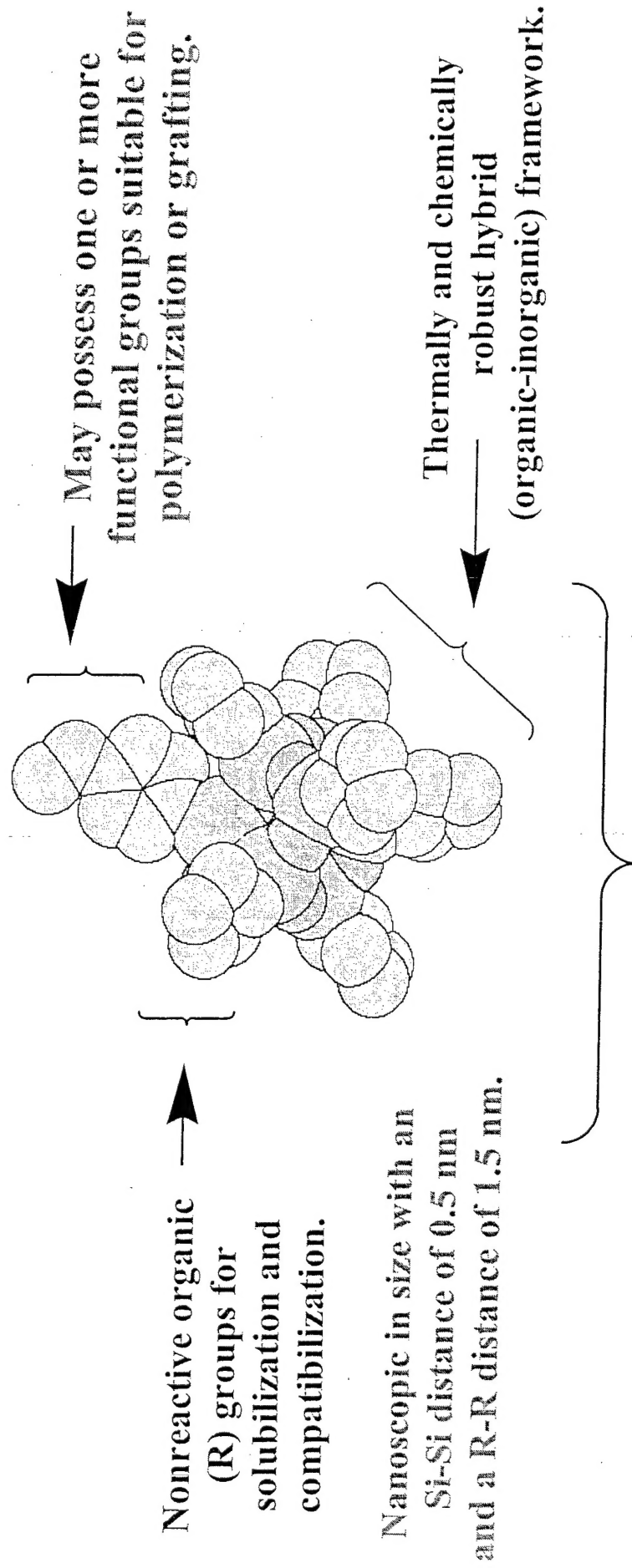
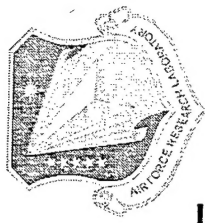
PAS-03-061

• Hybrid plastics bridge the differences between ceramics and polymers

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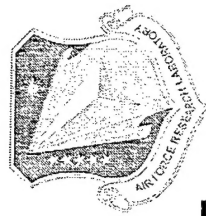
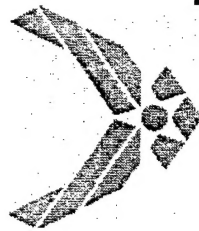
Anatomy of a POSS Nanostructure



Precise three-dimensional structure for molecular level reinforcement of polymer segments and coils.

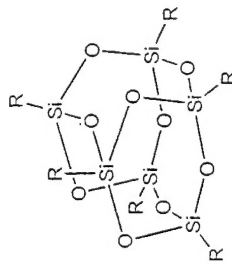
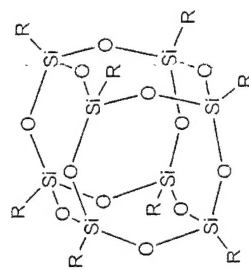
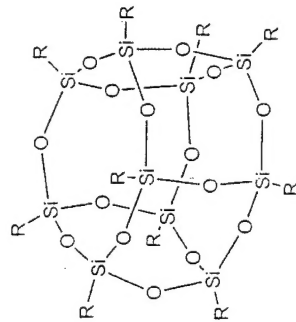
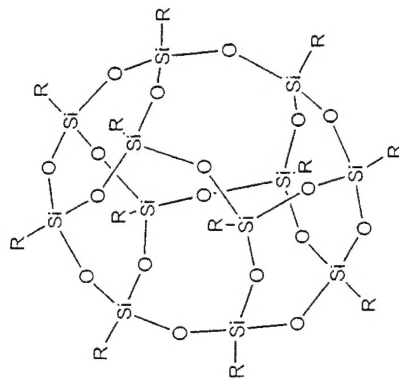
PAS-03-061

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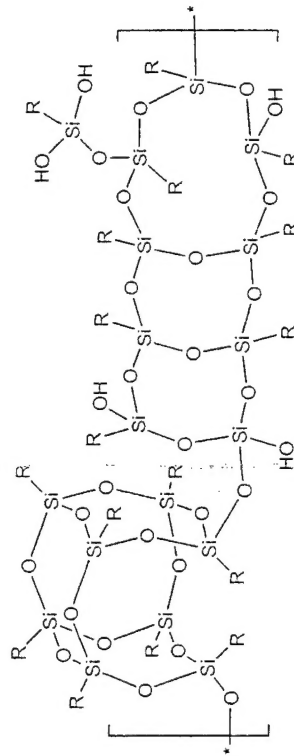


POSS Synthesis

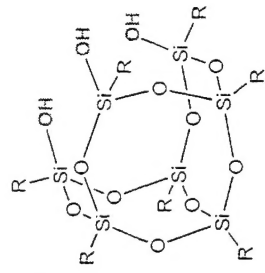
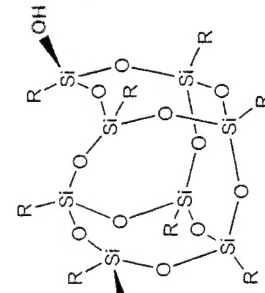
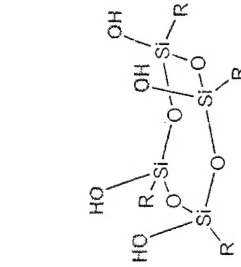
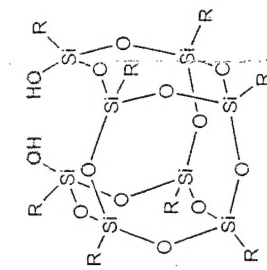
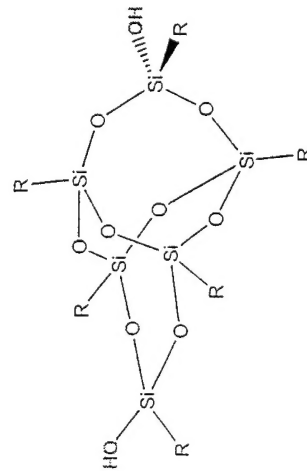
RSiX₃ acid or base hydrolysis



Completely
condensed



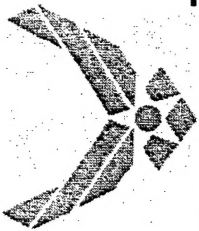
Resin



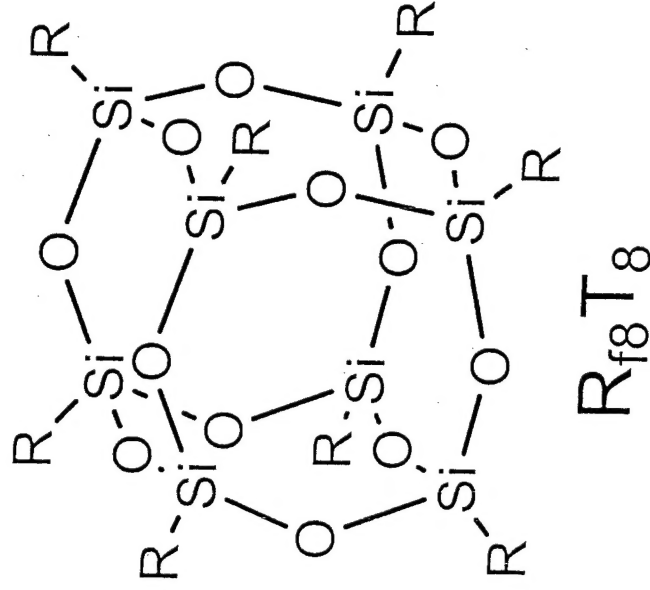
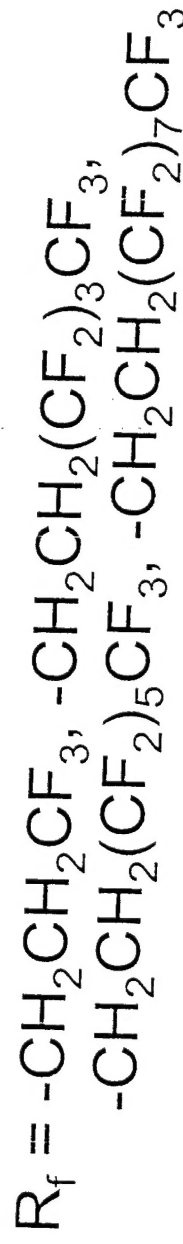
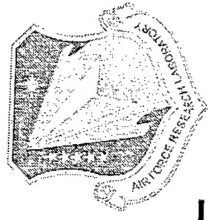
Incompletely condensed

Brown, Feher, AFRL, Hybrid Plastics

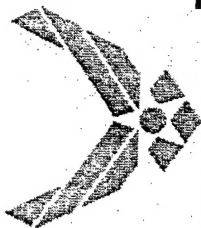
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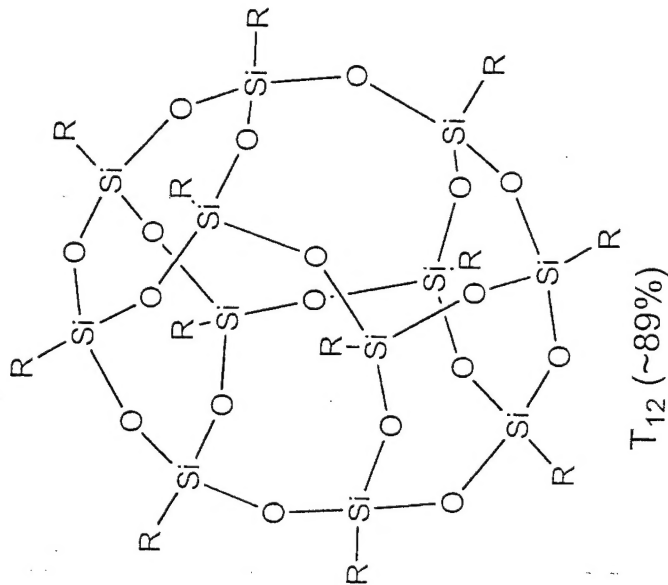
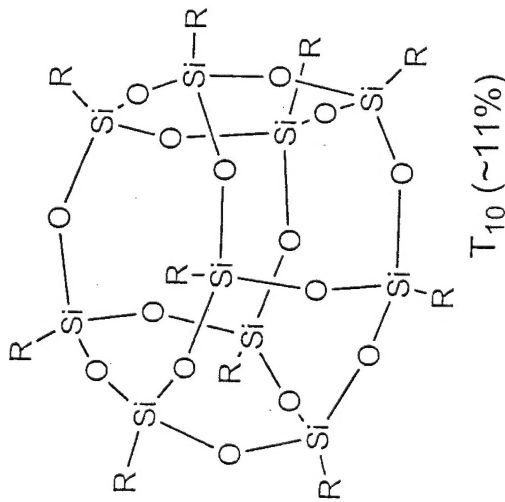
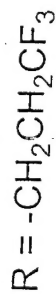
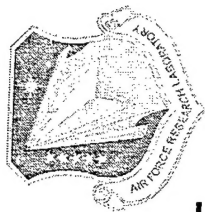
FluoroPOSS Synthesis



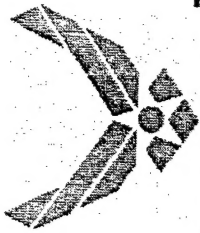
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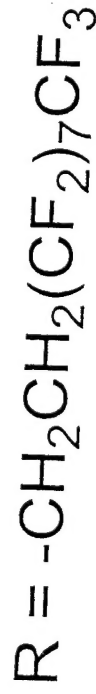
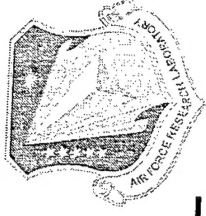
Fluoropropyl T_n



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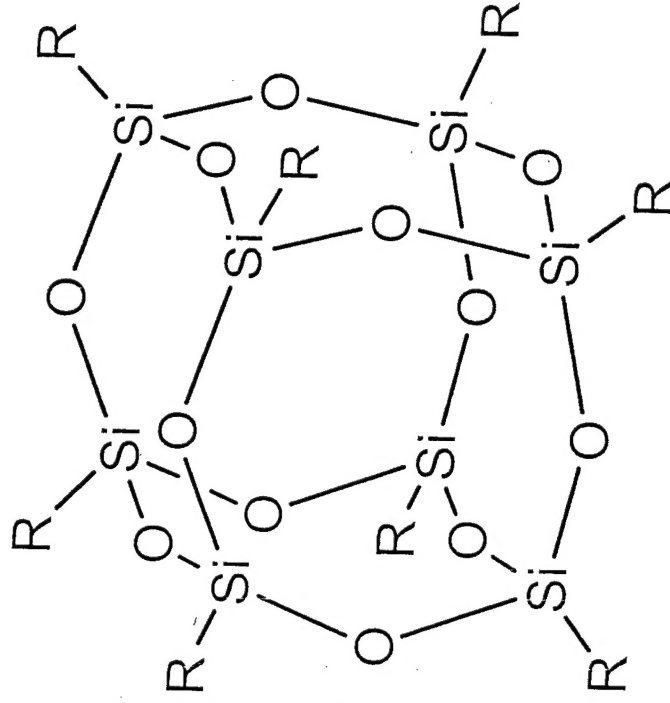


Fluorodecyl₈T₈

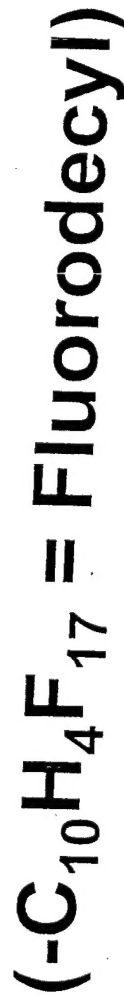


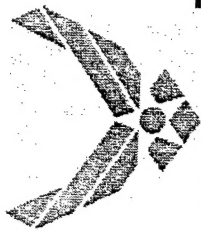
$$M_W = 3993.54 \text{ g/mol}$$

$$\rho = 2.058 \text{ g/mL}$$

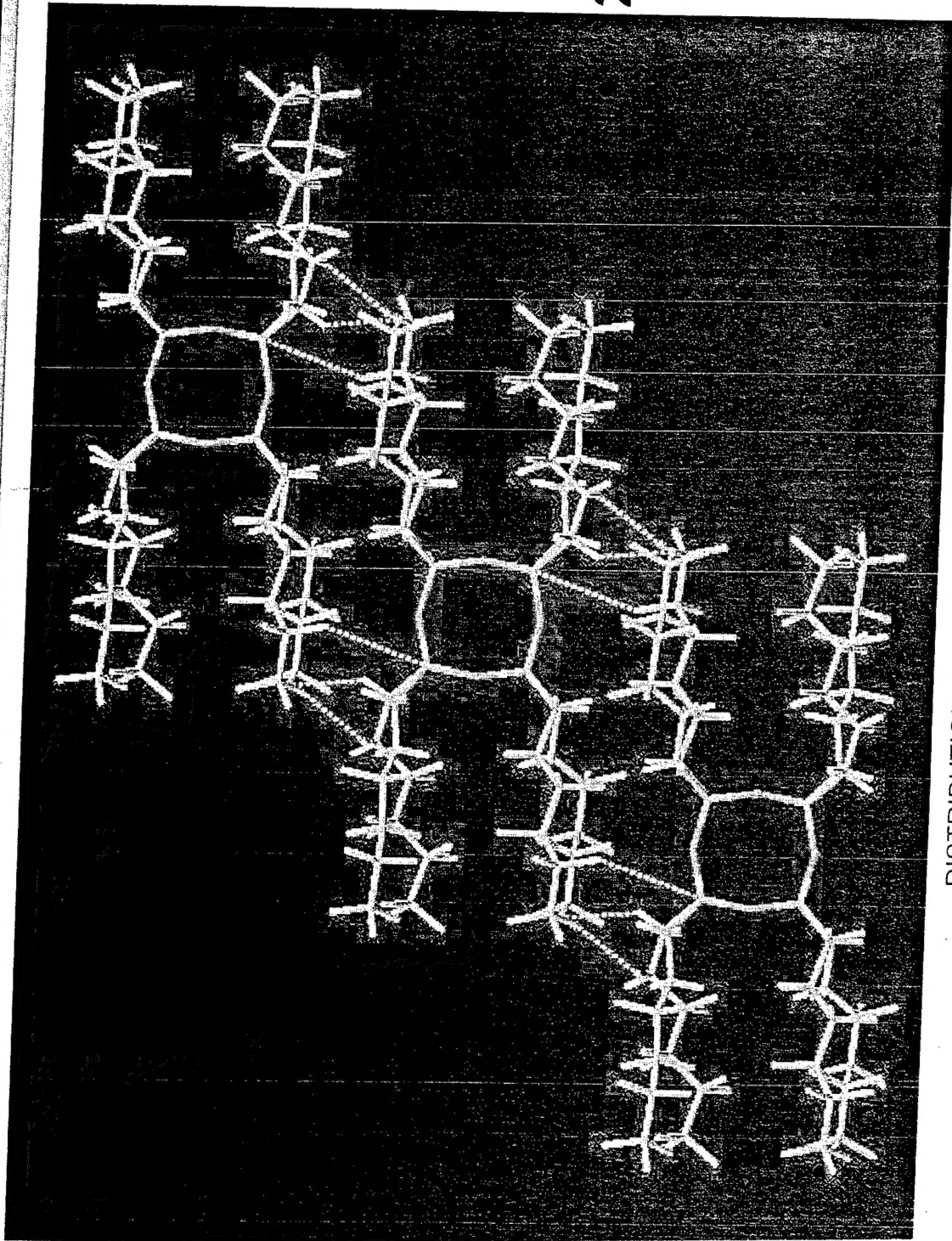
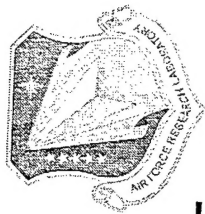


T₈





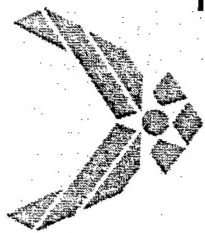
Fluorohexyl₈T₈



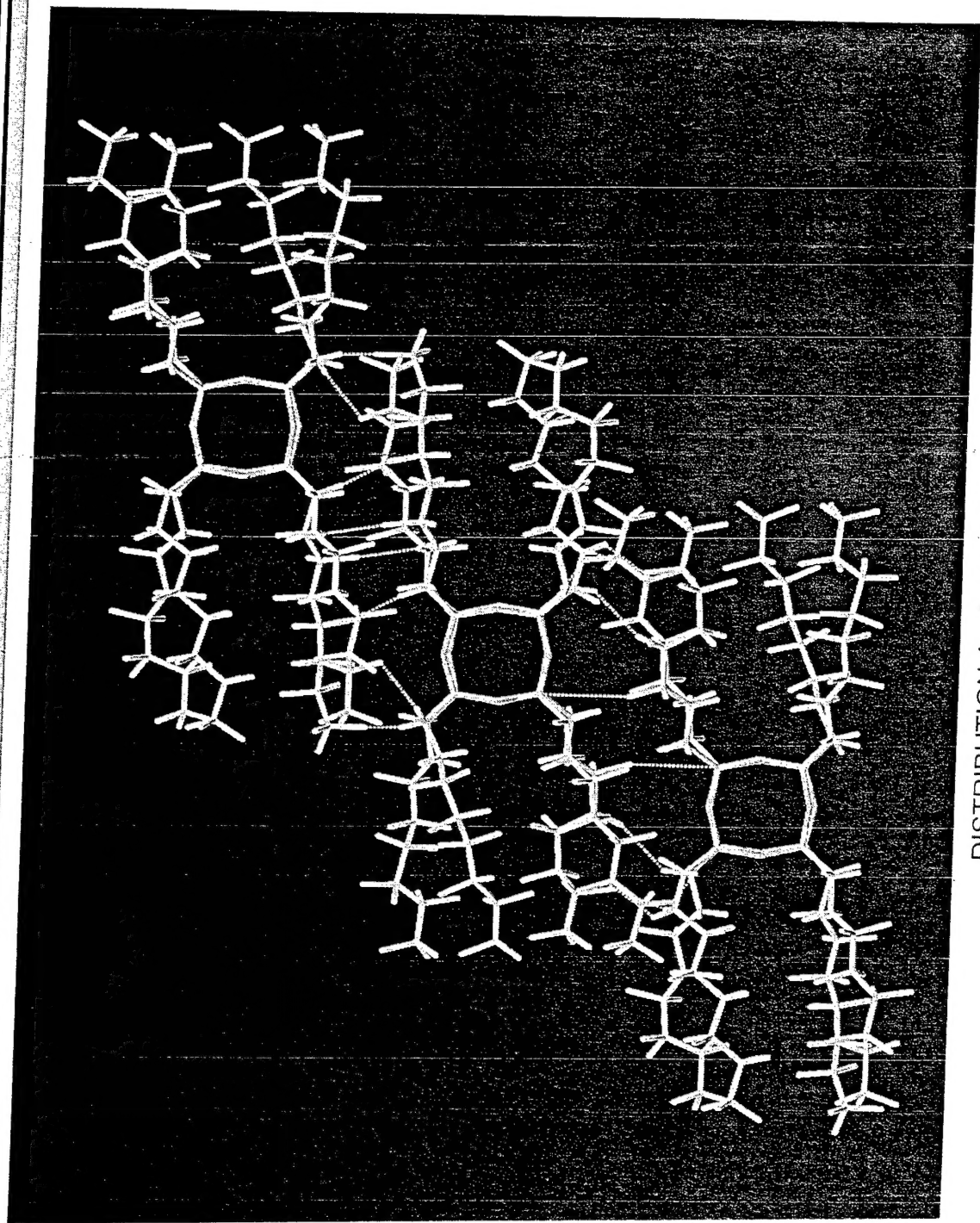
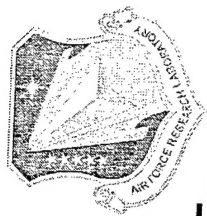
$$\rho = 1.98 \text{ g/mL}$$

$$M_w = 2393.33 \text{ g/mol}$$

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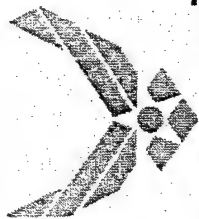
Fluorooctyl₈T₈



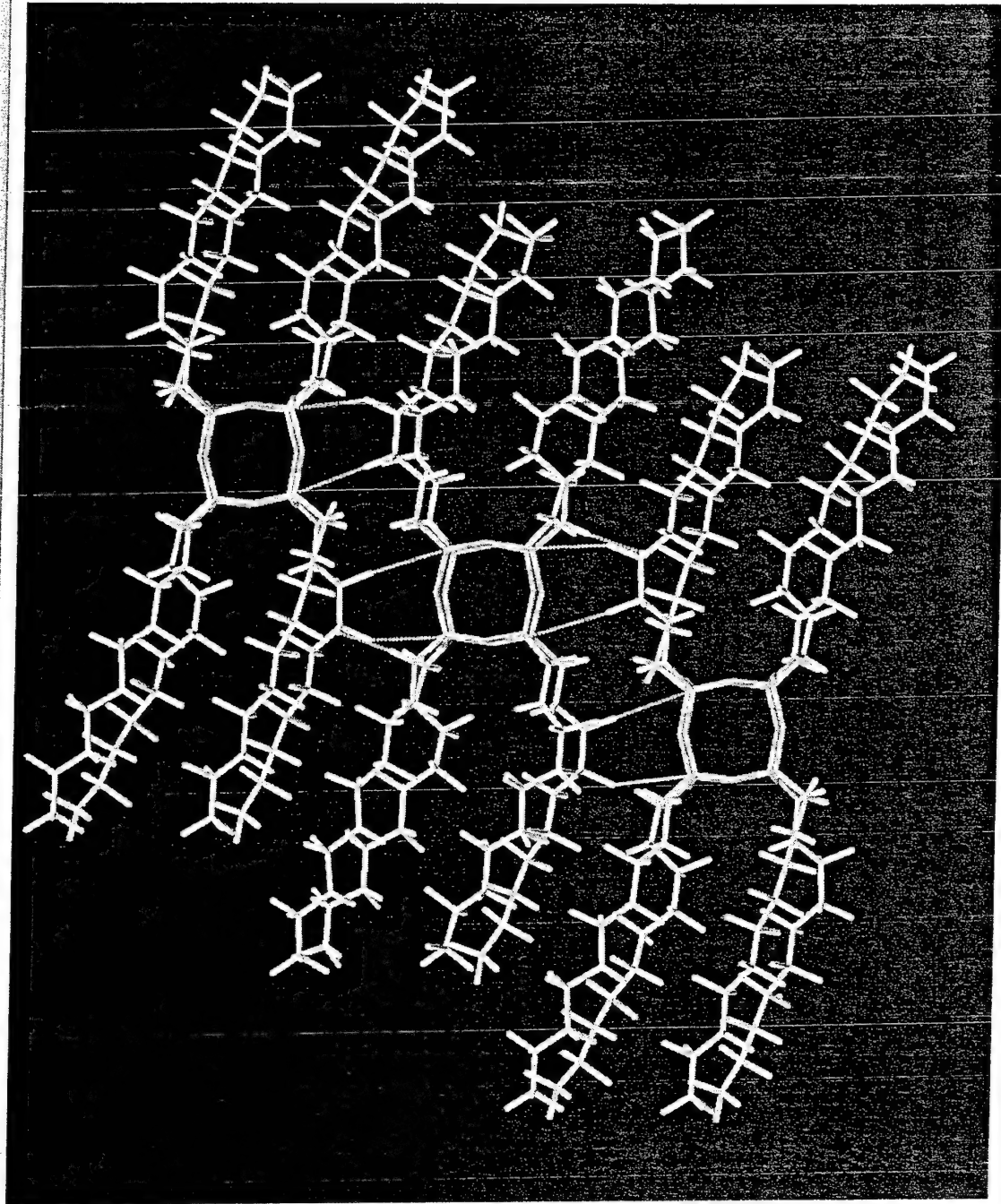
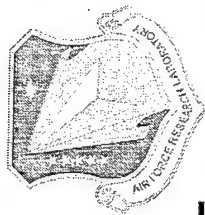
$$\rho = 2.05 \text{ g/mL}$$

$$M_w = 3193.45 \text{ g/mol}$$

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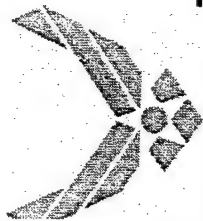
Fluorodecyl₈T₈



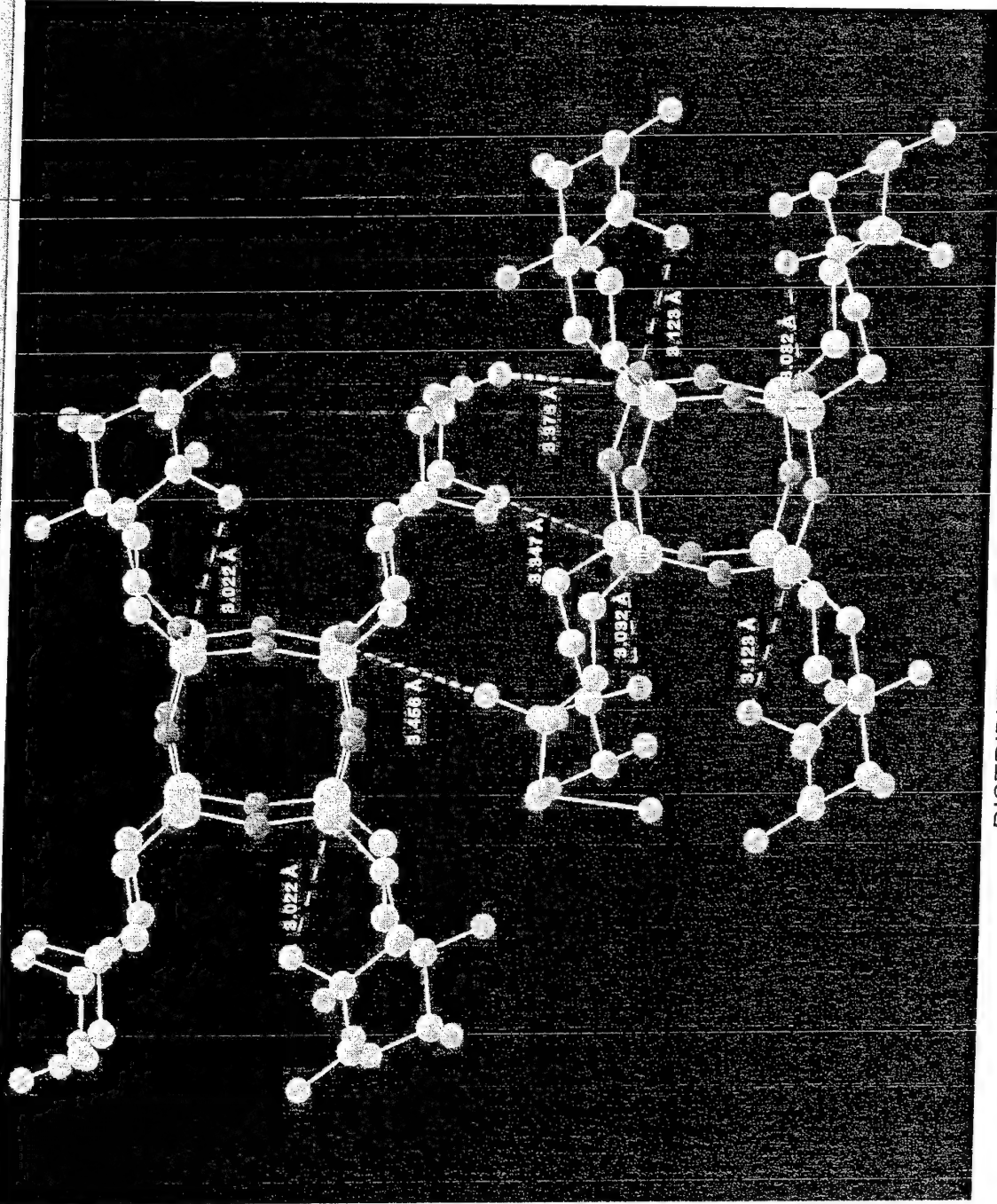
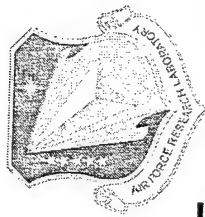
$$\rho = 2.06 \text{ g/mL}$$

$$M_w = 3993.54 \text{ g/mol}$$

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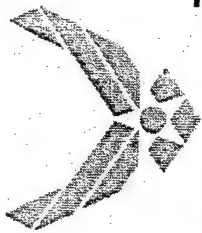


Fluorodecyl₈T₈

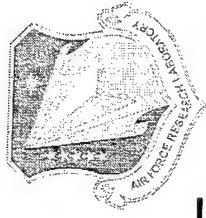


Inter- and intra-
molecular Si-F
contacts affect
crystal packing

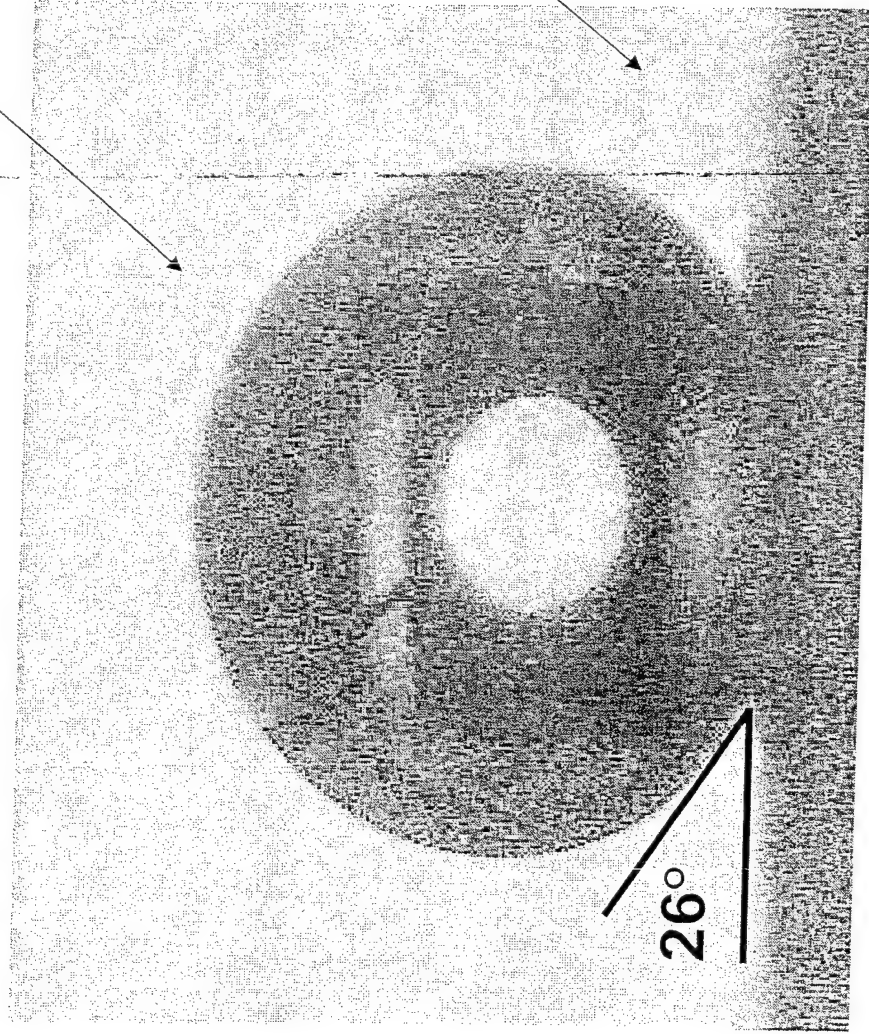
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Contact Angle of Water on Fluorodecyl POSS Surface



Drop of
 H_2O



POSS
Coated
Surface

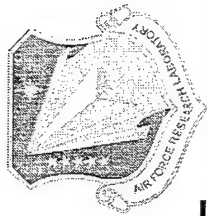
- Anti Icing surfaces
- Low Friction Surfaces

40° Higher than PTFE

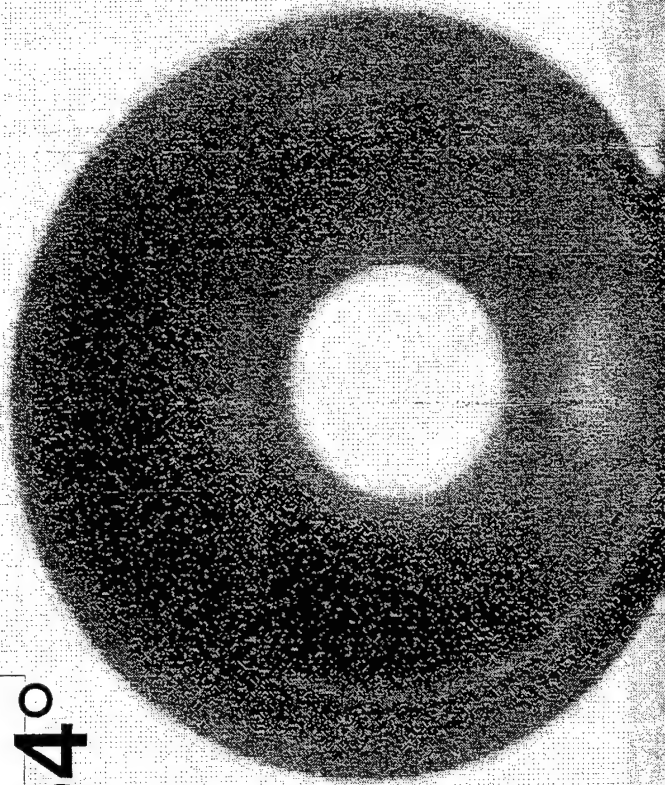
26°



Contact Angle of Water on Fluorodecyl POSS Surface



154°

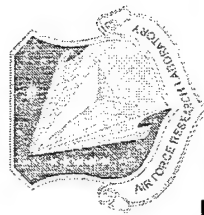


40° Higher than PTFE

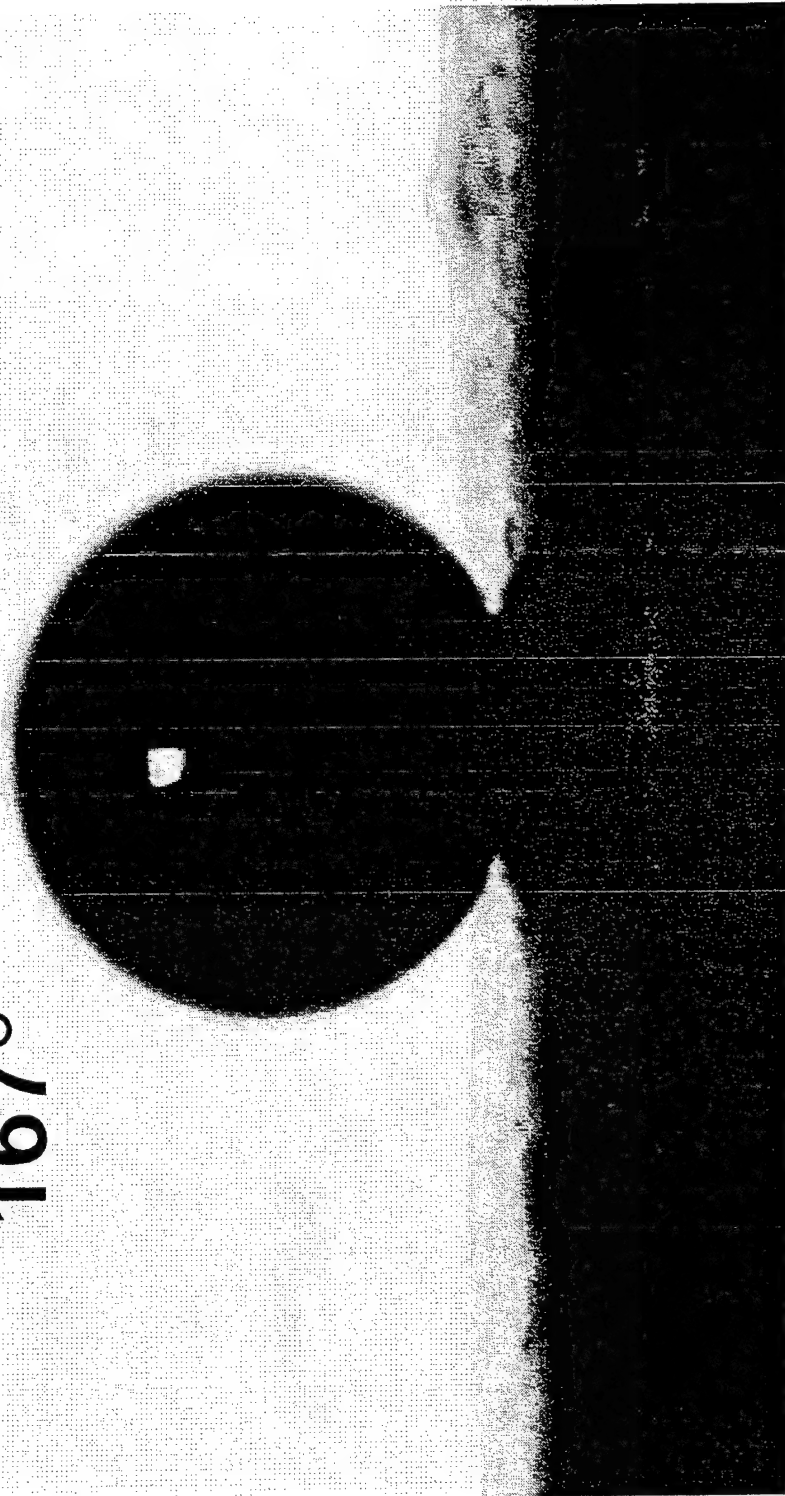
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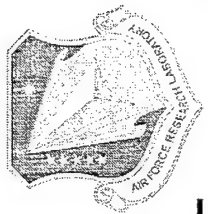
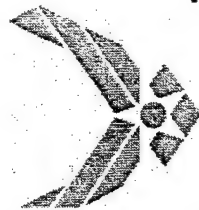
Contact Angle of Mercury on Fluorodecyl POSS Surface



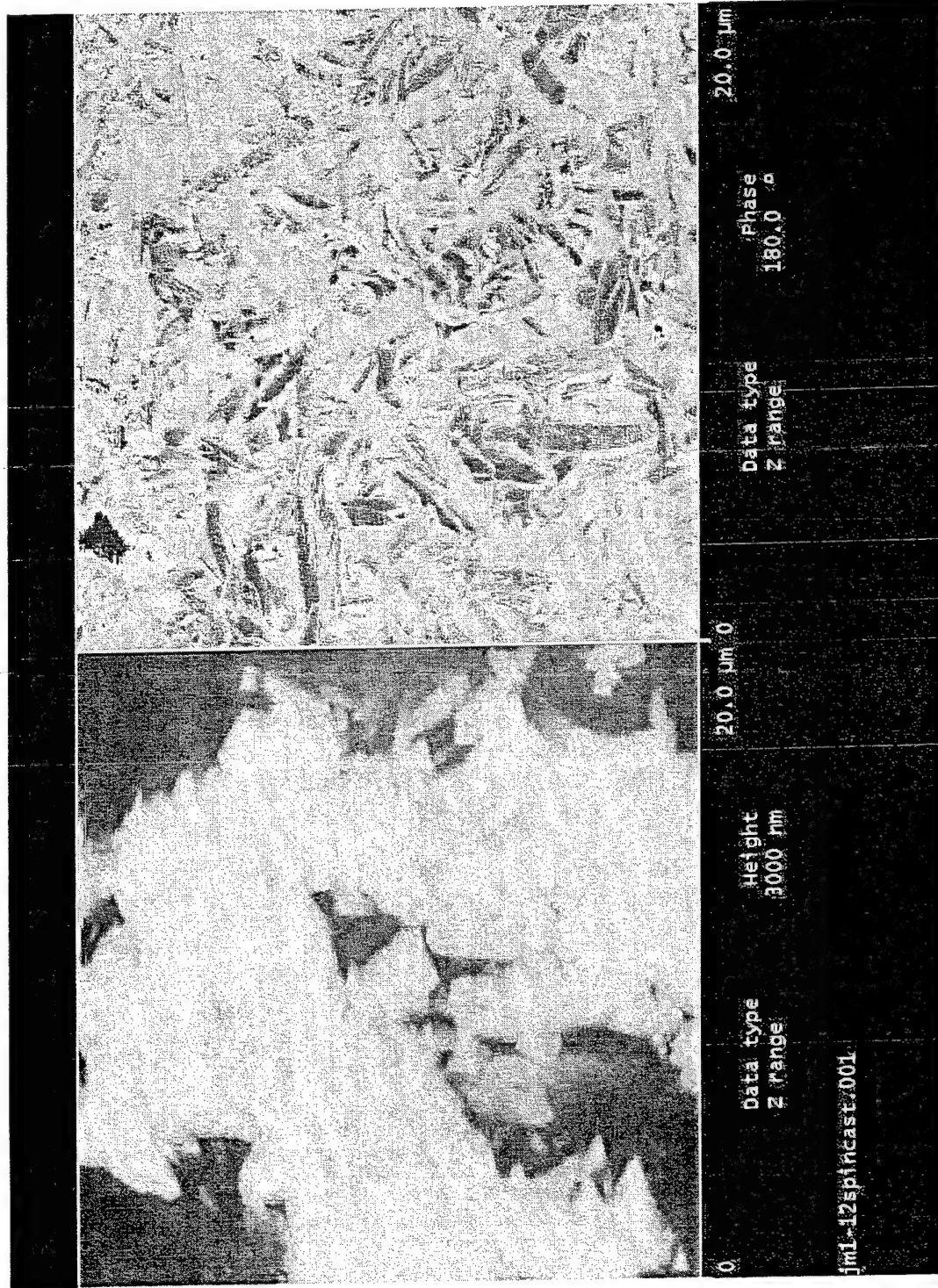
167°



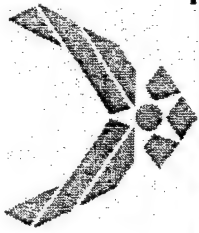
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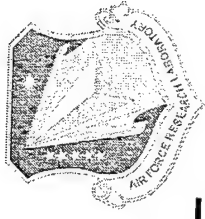
AFM Image of Spin-Cast Fluorodecyl₁₈T₈ Surface



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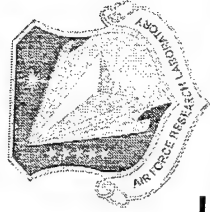
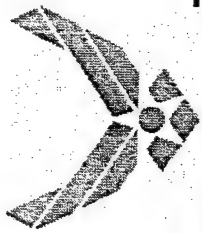
Surface Energy of Fluorosiloxanes



<u>Polymer</u>	<u>Surface Energy (mJ/m²)</u>
Poly(methylheptadecafluorodecylsiloxane)	7.0
Poly(methylnonafluorohexylsiloxane)	9.5
Poly(methyltrifluoropropylsiloxane)	13.6
Poly(dimethylsiloxane) (PDMS)	22.8
Poly(tetrafluoroethylene) (PTFE)	19.1

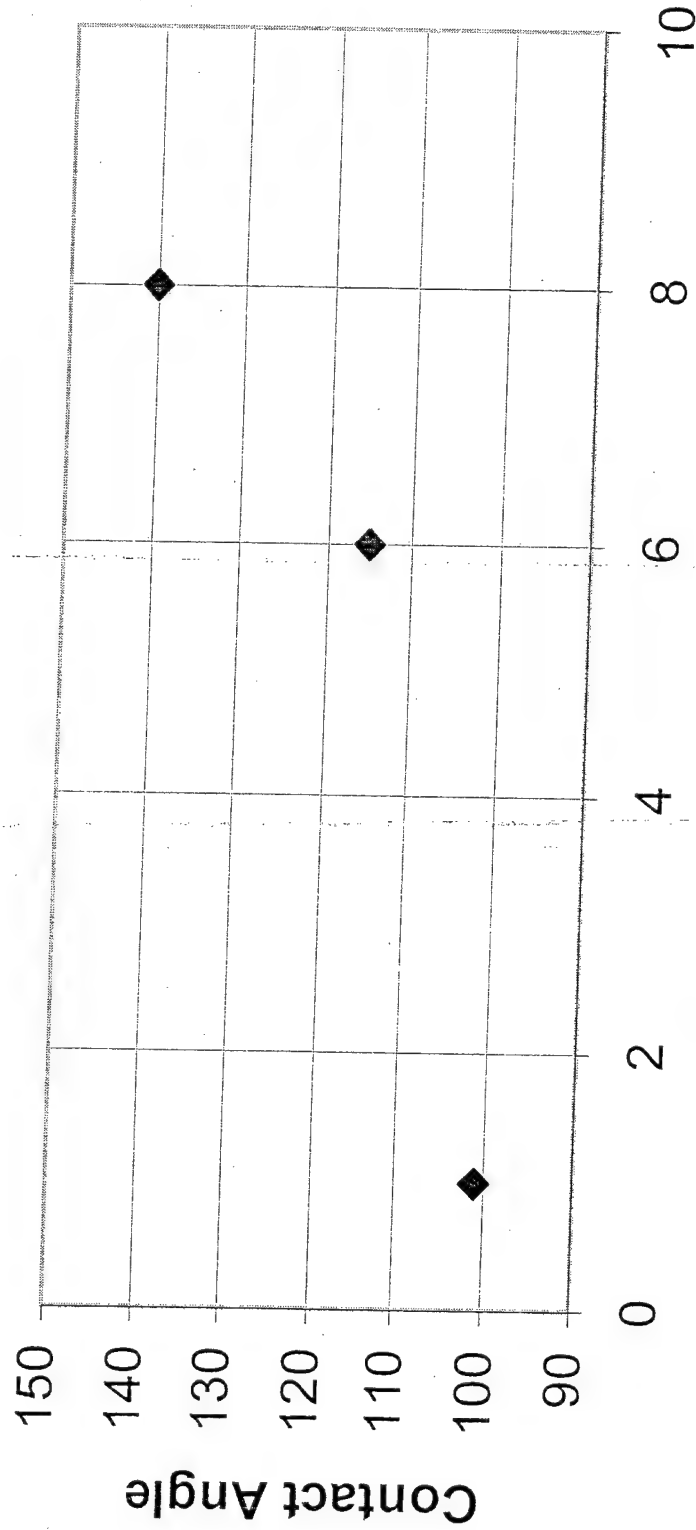
Maxson, M. T.; Norris, A. W.; Owen, M. J. "Fluorosilicones" In "Modern Fluoropolymers"; Scheirs, J. Ed.; J. Wiley & Sons: New York, 1997, pp 359-372.

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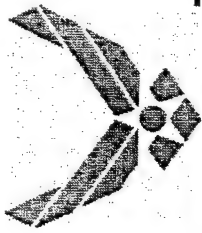


Contact Angle and Chain Length

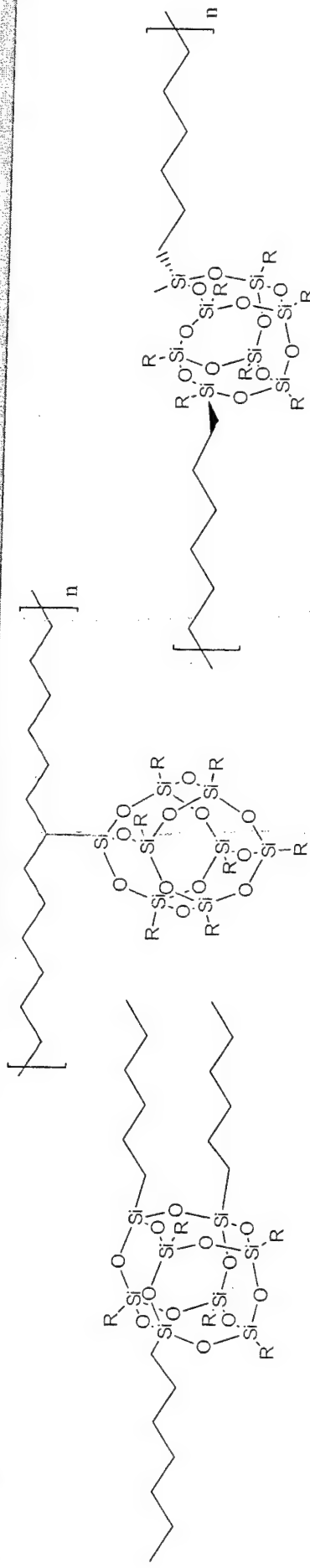
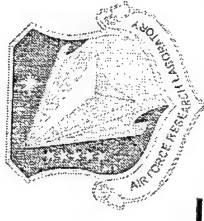
Chain Length vs. Contact Angle



Number of Fluorocarbon Atoms



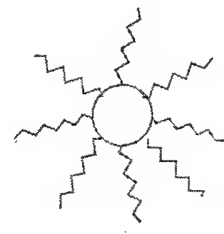
POSS Polymer Incorporation



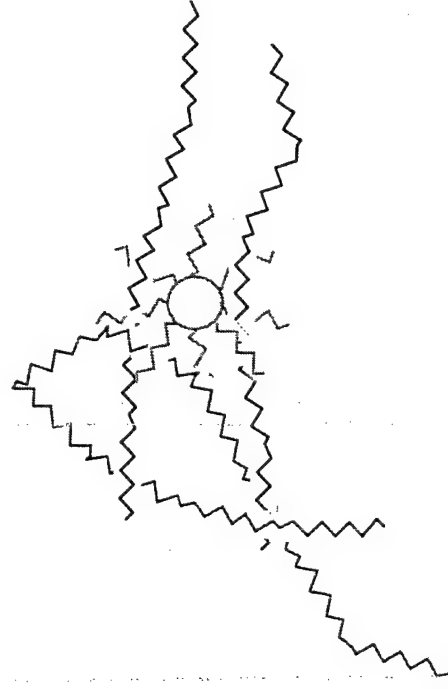
Cross-linker

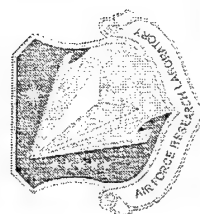
POSS Pendant

Bead Copolymer

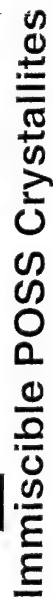
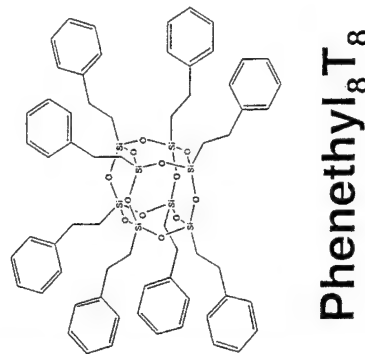
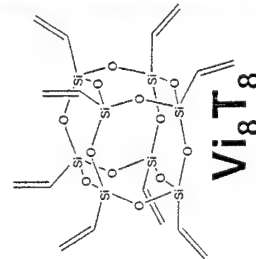
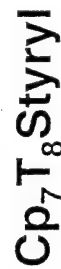
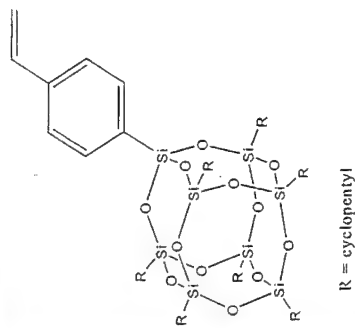


POSS Blending

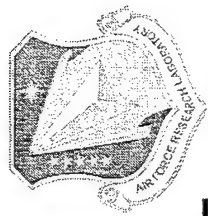




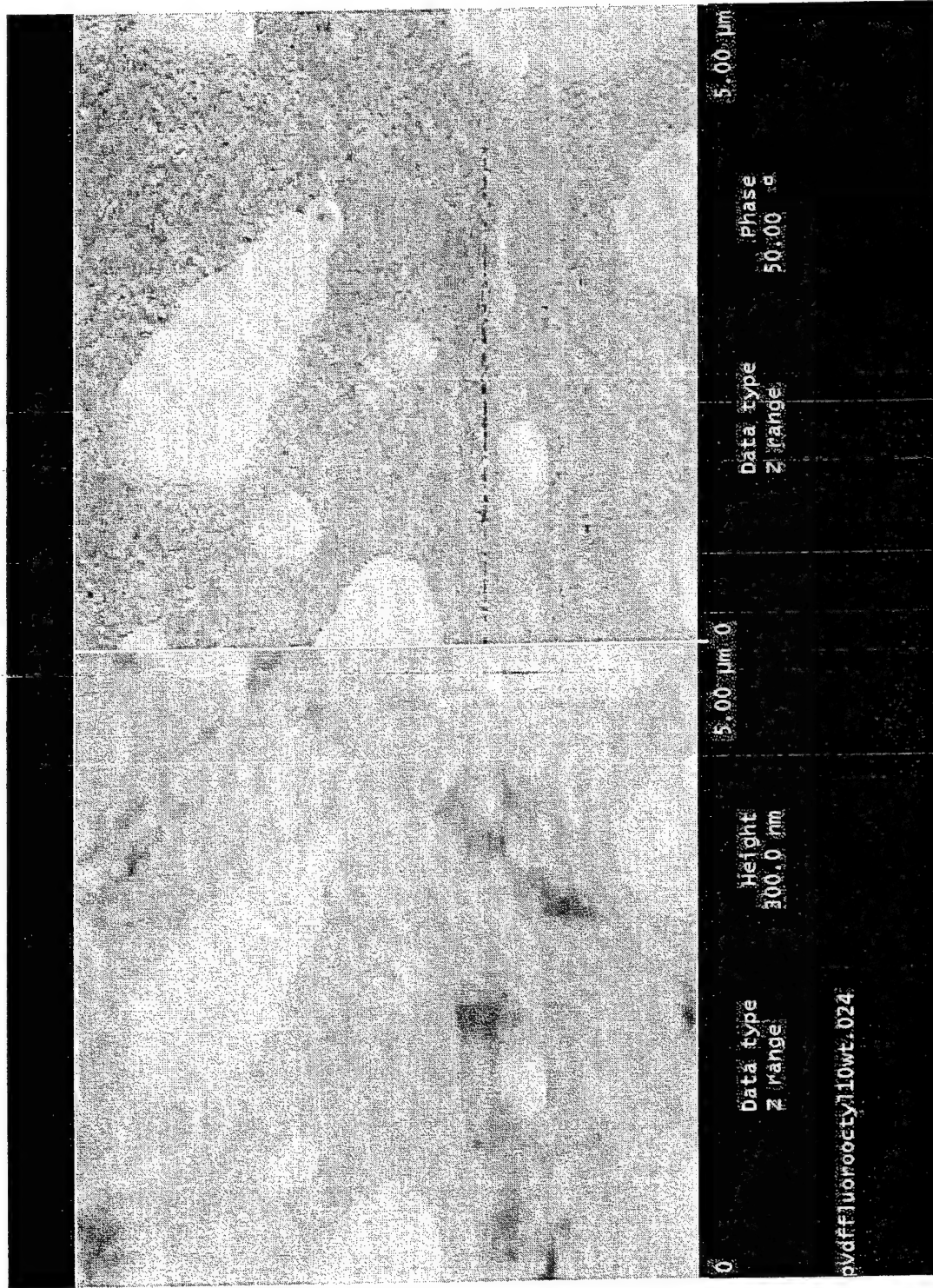
50 Wt % POSS Blends in 2 Million MW PS

Blanski, et al., *Polymer Preprints*, **2000**, 41(1): p. 585.

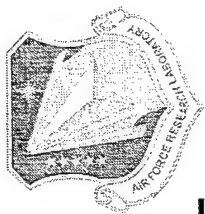
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1. *Chlorophyll a* (Chl *a*)
 2. *Chlorophyll b* (Chl *b*)
 3. *Chlorophyll c* (Chl *c*)
 4. *Chlorophyll d* (Chl *d*)
 5. *Chlorophyll e* (Chl *e*)
 6. *Chlorophyll f* (Chl *f*)
 7. *Chlorophyll g* (Chl *g*)
 8. *Chlorophyll h* (Chl *h*)
 9. *Chlorophyll i* (Chl *i*)
 10. *Chlorophyll j* (Chl *j*)
 11. *Chlorophyll k* (Chl *k*)
 12. *Chlorophyll l* (Chl *l*)
 13. *Chlorophyll m* (Chl *m*)
 14. *Chlorophyll n* (Chl *n*)
 15. *Chlorophyll o* (Chl *o*)
 16. *Chlorophyll p* (Chl *p*)
 17. *Chlorophyll q* (Chl *q*)
 18. *Chlorophyll r* (Chl *r*)
 19. *Chlorophyll s* (Chl *s*)
 20. *Chlorophyll t* (Chl *t*)
 21. *Chlorophyll u* (Chl *u*)
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 23. *Chlorophyll w* (Chl *w*)
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 25. *Chlorophyll y* (Chl *y*)
 26. *Chlorophyll z* (Chl *z*)
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 42. *Chlorophyll ap* (Chl *ap*)
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 130. *Chlorophyll axz* (Chl *axz*)
 131. *Chlorophyll ayz* (Chl *ayz*)
 132. *Chlorophyll ayz* (Chl *ayz*)
 133.



DISTRIBUTION A. Approved for public release; distribution unlimited.



0 1.00 μm 0

Data type
Z range

Height
50.00 nm

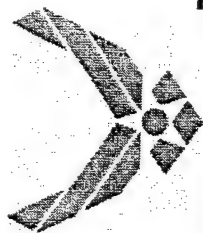
0 1.00 μm 100.0 μm

Data type
Z range

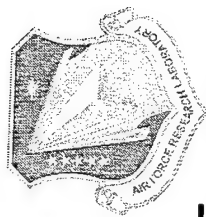
Phase
100.0 μm

0vdffLfpnt2p84wt.019

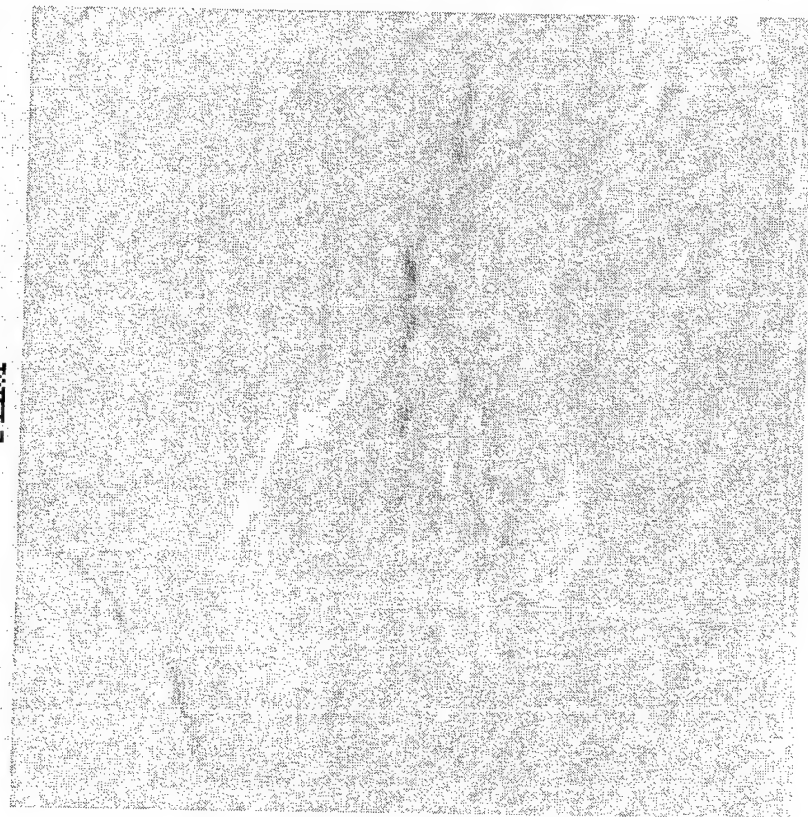
DISTRIBUTION A. Approved for public release; distribution unlimited.



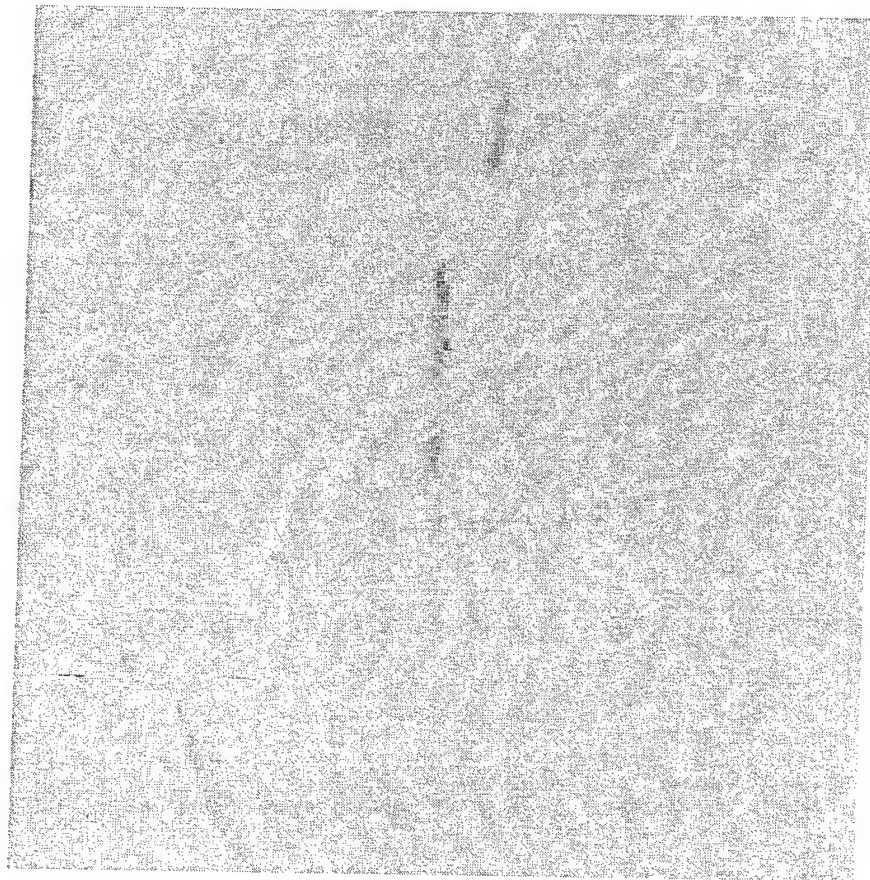
PVDF/Fluoropropyl_nT_n POSS



SEM



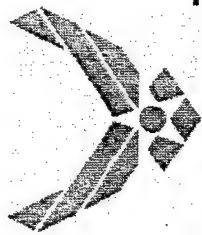
SEM Image



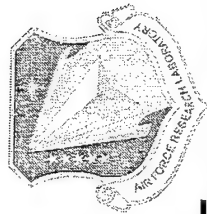
Carbon Map

SEM Image taken on cross-section of 1/4 inch thick sample bar.

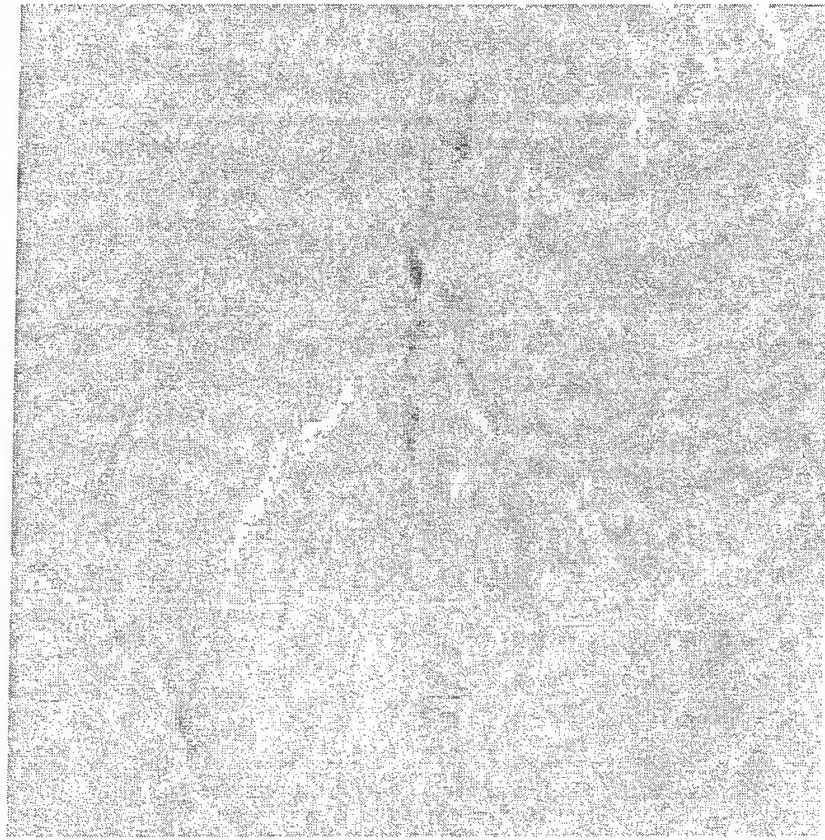
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PVDF/Fluoropropyl_nT_n POSS



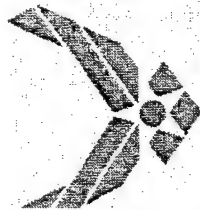
Fluorine Map



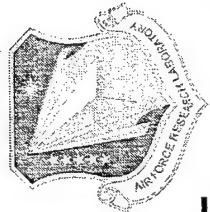
Silicon Map

Silicon map shows reasonable dispersion of POSS in polymer.

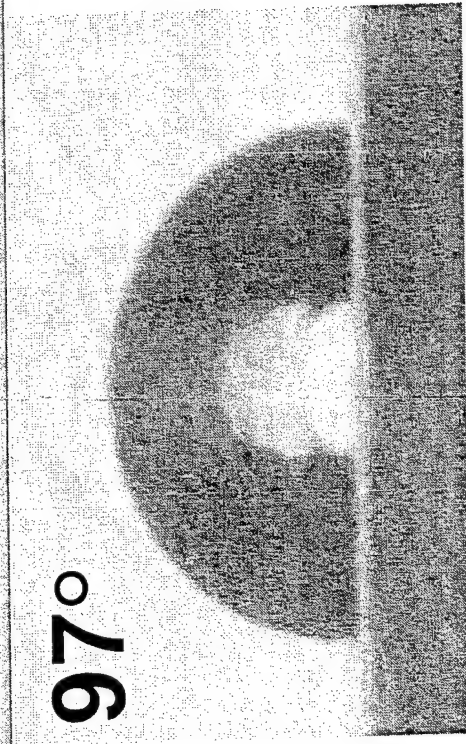
DISTRIBUTION A. Approved for public release; distribution unlimited.



Fluorinated Ethylene/Propylene

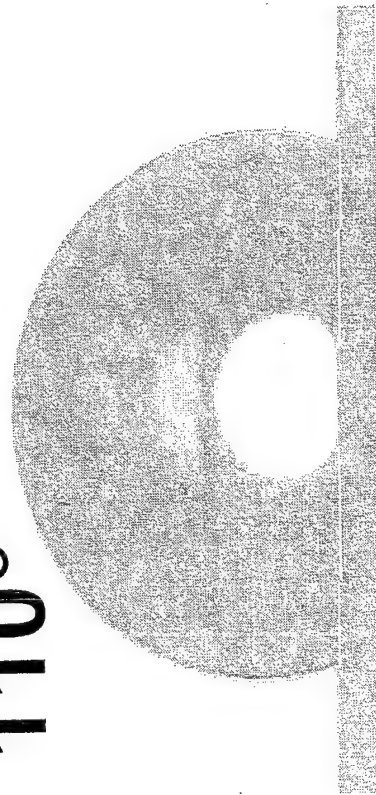


97°



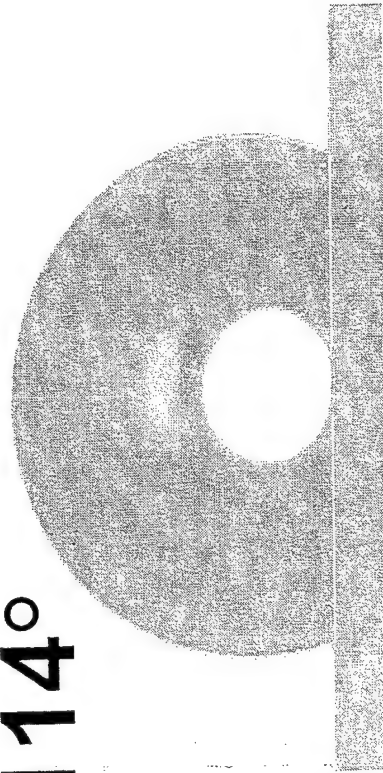
FEP

110°



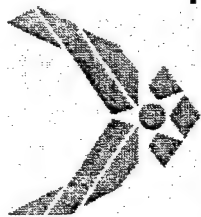
15% FO₈T₈

114°

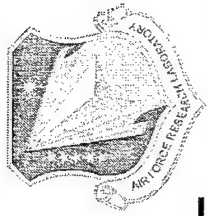


15% FD₈T₈

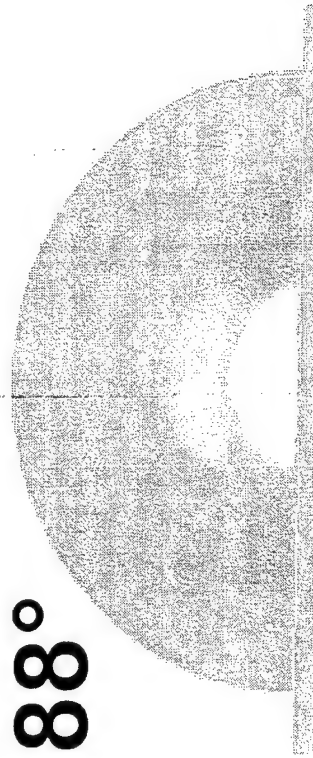
DISTRIBUTION A. Approved for public release; distribution unlimited.



Poly(chlorotrifluoroethylene)

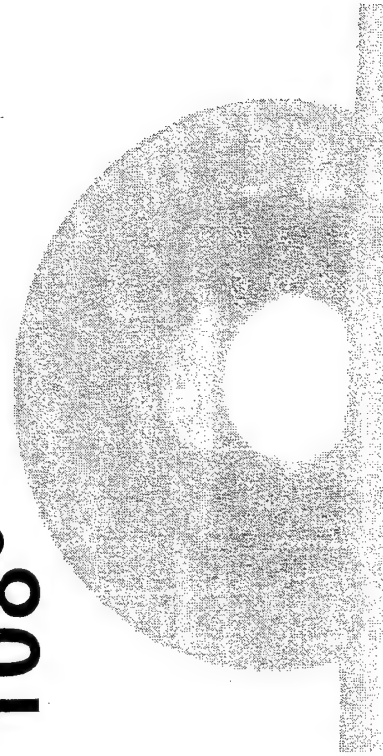


88°



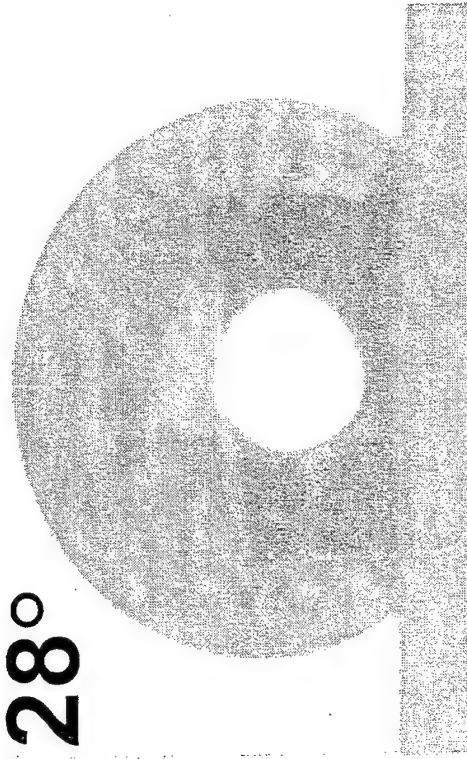
PCTFE

108°



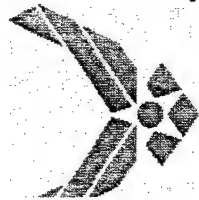
10% FO₈T₈

128°

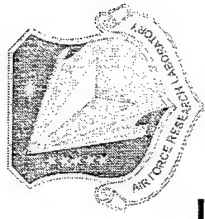


10% FD₈T₈

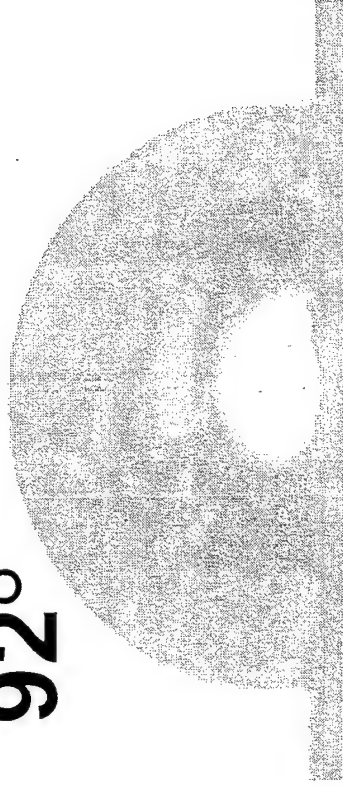
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Amorphous FEP

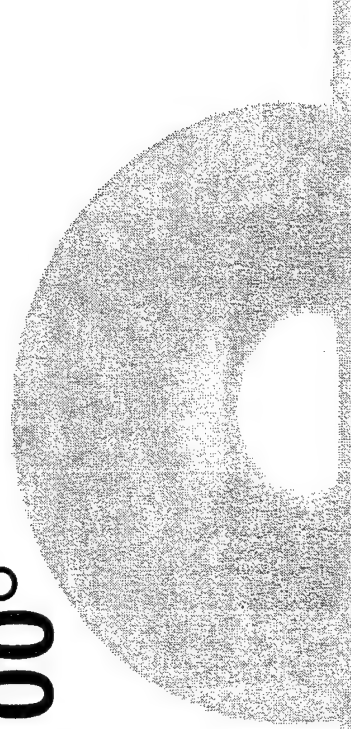


92°



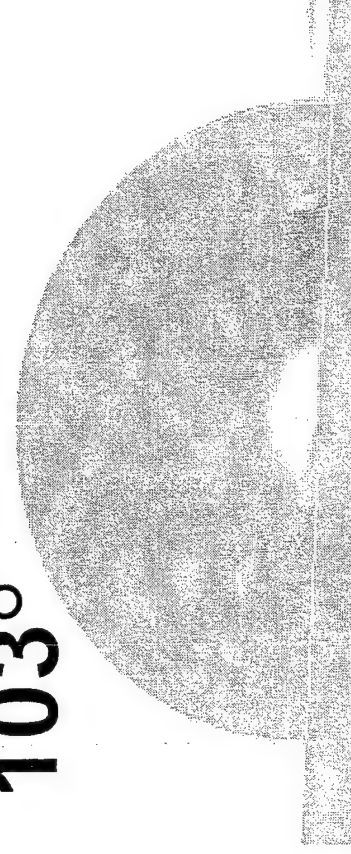
AFEP

100°



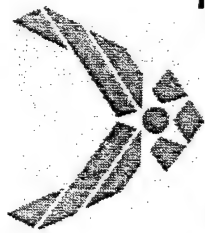
AFEP with 10% FO₈T₈

103°

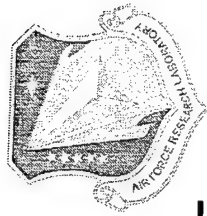


AFEP with 10% FD₈T₈

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Water Contact Angle



Polymer	No POSS	FO ₈ T ₈	FD ₈ T ₈
PCTFE	88°	108°	128°
FEP	97°	110°	114°
Amor. FEP	92°	100°	103°

Fluoropropyl POSS (FP_nT_n)

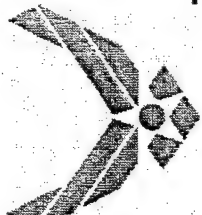
Fluorooctyl POSS (FO₈T₈)

Fluorodecyl POSS (FD₈T₈)

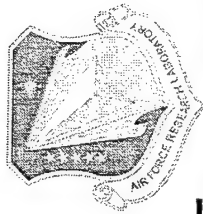
101°

115°

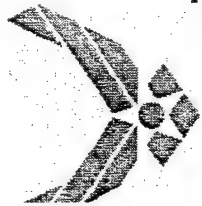
154°



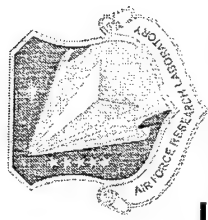
Summary



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Acknowledgements

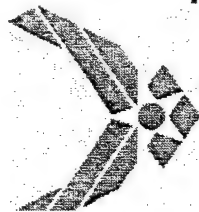


Assistance from others at AFRL:

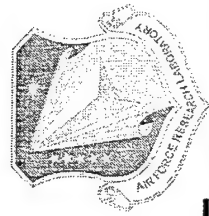
Crystal Structures
SEM Images
SEM Images
AFM Images

Ashwani Vij
Marietta Fernandez
Erik Weber
Brian Moore

Financial Support:
Air Force Office of Scientific Research
Air Force Research Laboratory, Propulsion Directorate

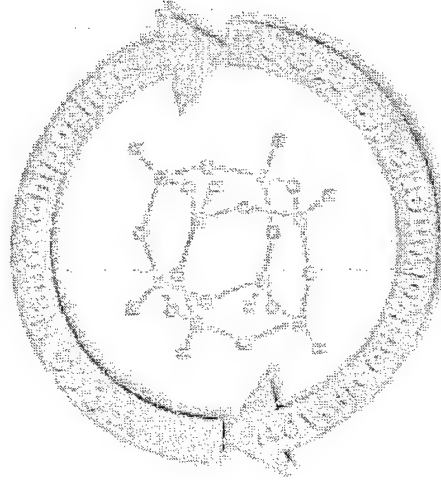


Acknowledgements



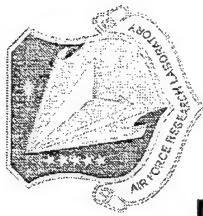
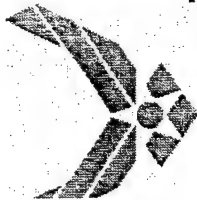
The Polymer Working Group at Edwards Air Force Base is:

Maj Connie Schlaefer
Mr. Pat Ruth
Dr. Sandra Tomczak
Mr. Brian Moore
Dr. Brent Viers
Dr. Darrell Marchant
Lt Will Cooper
Mr. Scott Barker



Dr. Shawn Phillips
Lt Amy Palacek
Dr. Rusty Blanski
Dr. Joe Mabry
Mrs. Sherly Largo
Dr. Tim Haddad
Lt Laura Moody

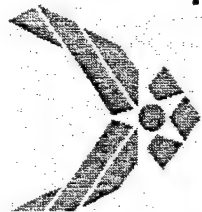
Financial Support:
Air Force Office of Scientific Research
Air Force Research Laboratory, Propulsion Directorate



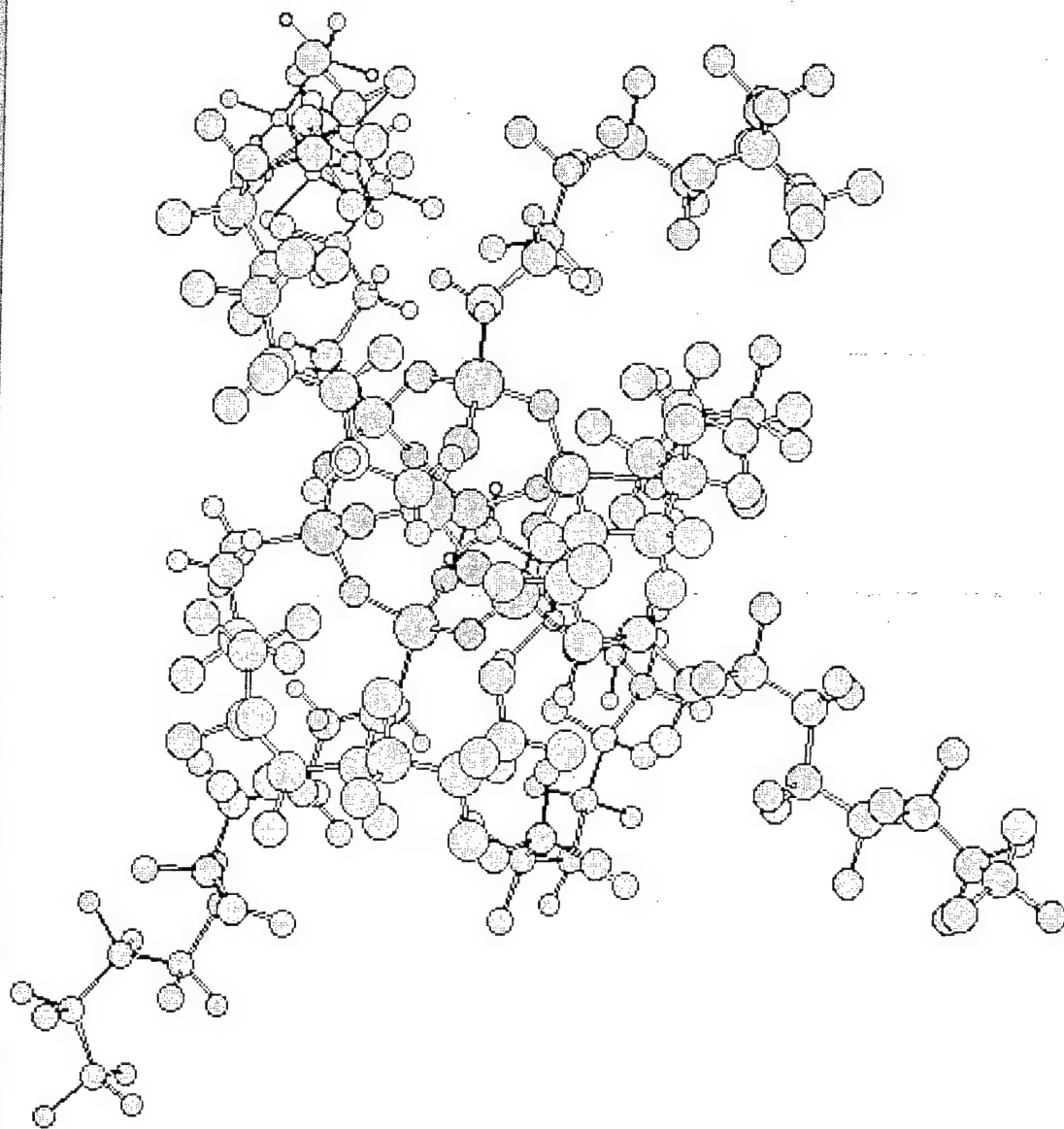
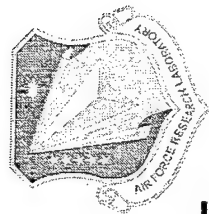
Backup Slides

PAS-03-061

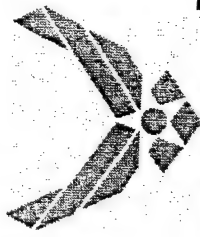
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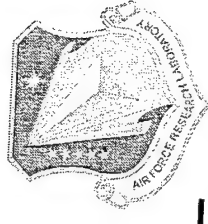
Gas Phase Model of Fluorodecyl₈T₈



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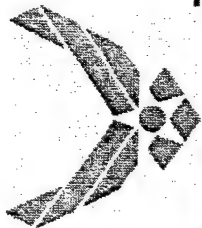


Density

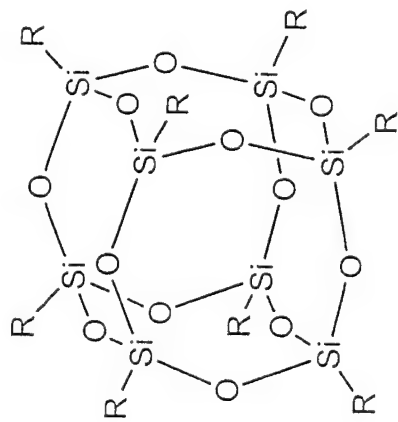
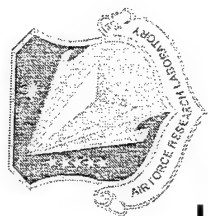


<u>Compound</u>	<u>Density (g/mL)</u>
• PVDF	1.75-1.78
• PCTFE	2.08-2.19
• FEP	2.12-2.17
• Fluoropropyl POSS	1.59
• Fluorohexyl POSS (crystal)	1.86 (1.98)
• Fluorooctyl POSS (crystal)	1.88 (2.05)
• Fluorodecyl POSS (crystal)	1.95 (2.06)

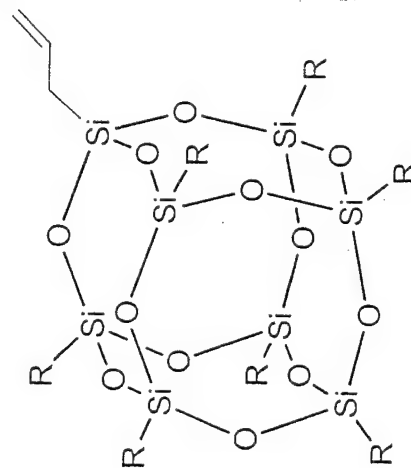
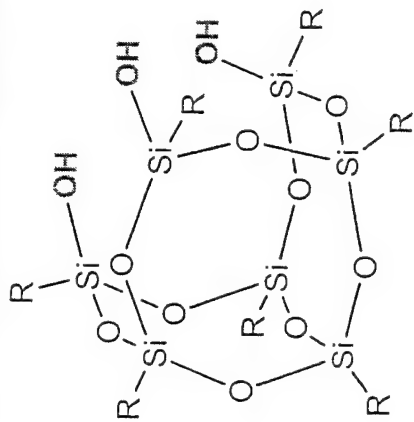
DISTRIBUTION A. Approved for public release; distribution unlimited.



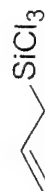
Fluorinated POSS Trisilanol



$\xrightarrow[\text{reflux}]{\text{Et}_4\text{NOH}}$

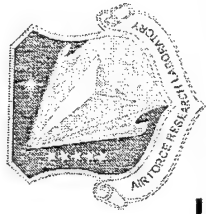
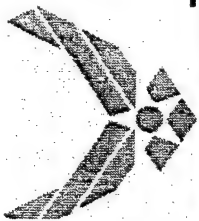


Et_3N

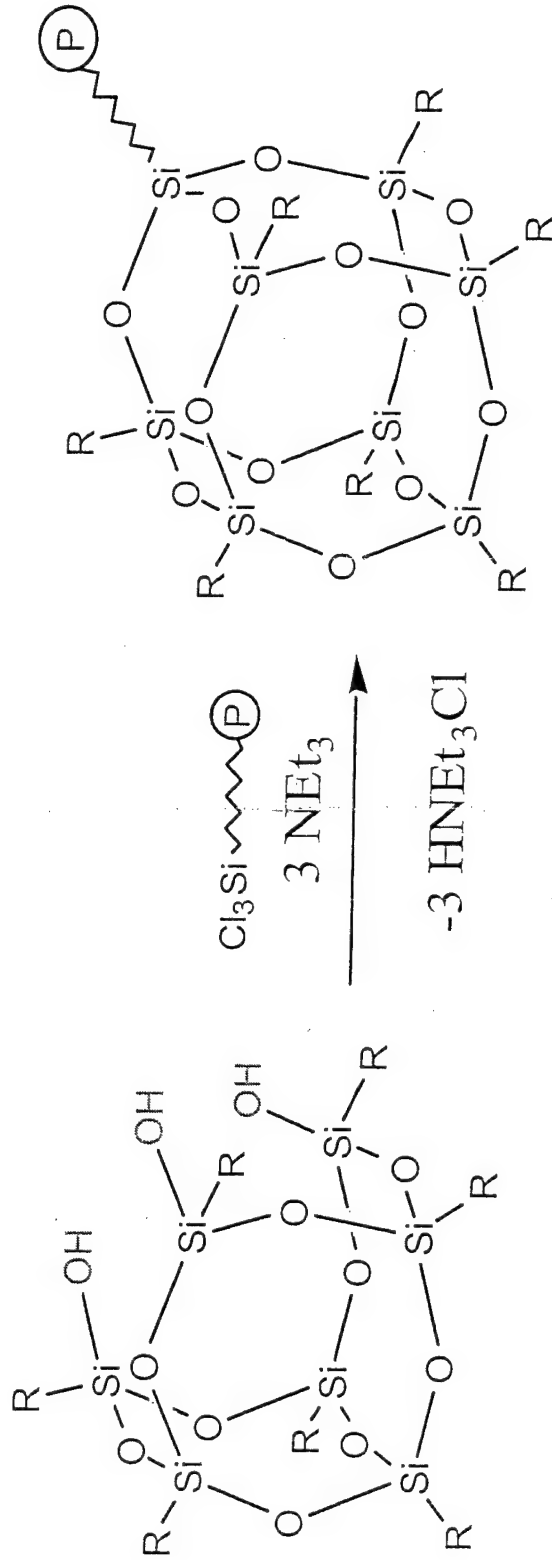


(or other chlorosilanes)

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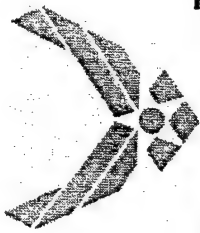
POSS Macromers for Nanocomposites



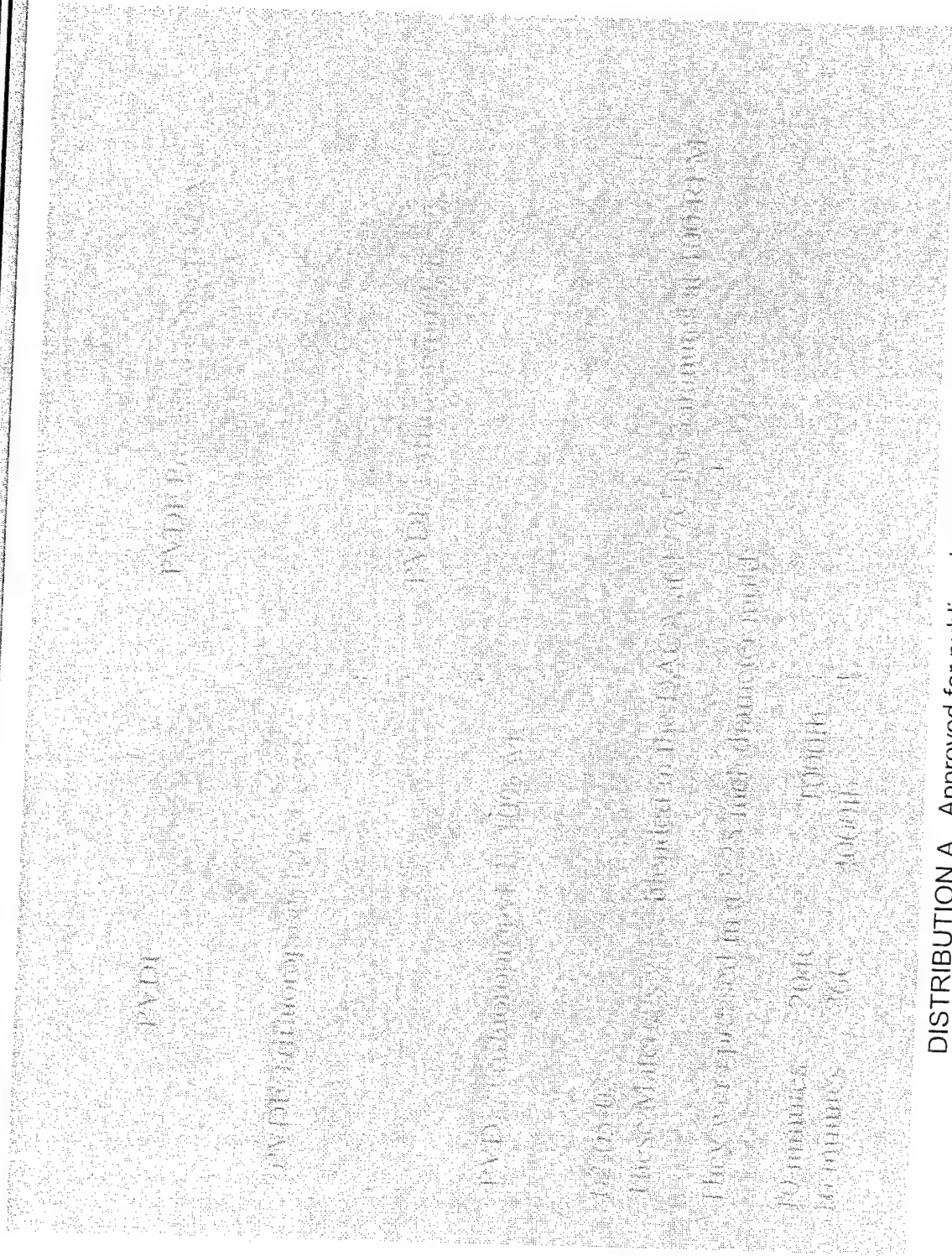
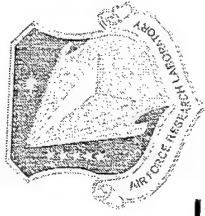
POSS-based macromers are available through either **Gelest** or **Aldrich**
POSS technology is commercialized by **Hybrid Plastics** in Fountain Valley CA

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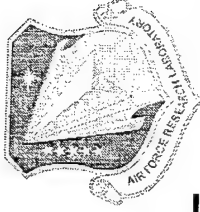
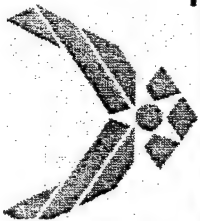
DISTRIBUTION A. Approved for public release; distribution unlimited.



PVDF Fluoropropyl_nT_n Blends



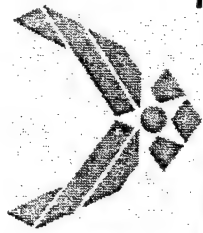
DISTRIBUTION A. Approved for public release; distribution unlimited.



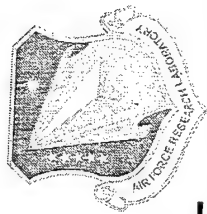
POSS Siloxanes

PAS-03-061

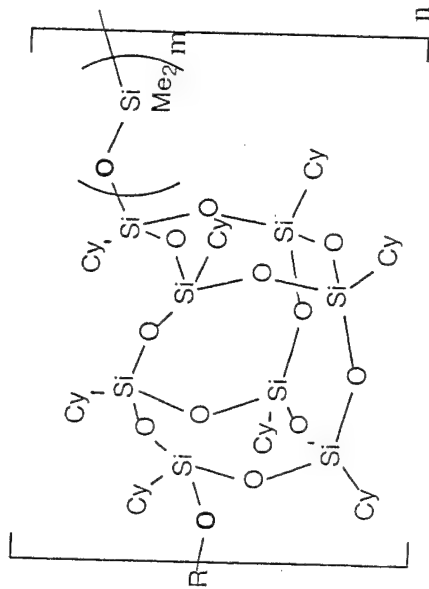
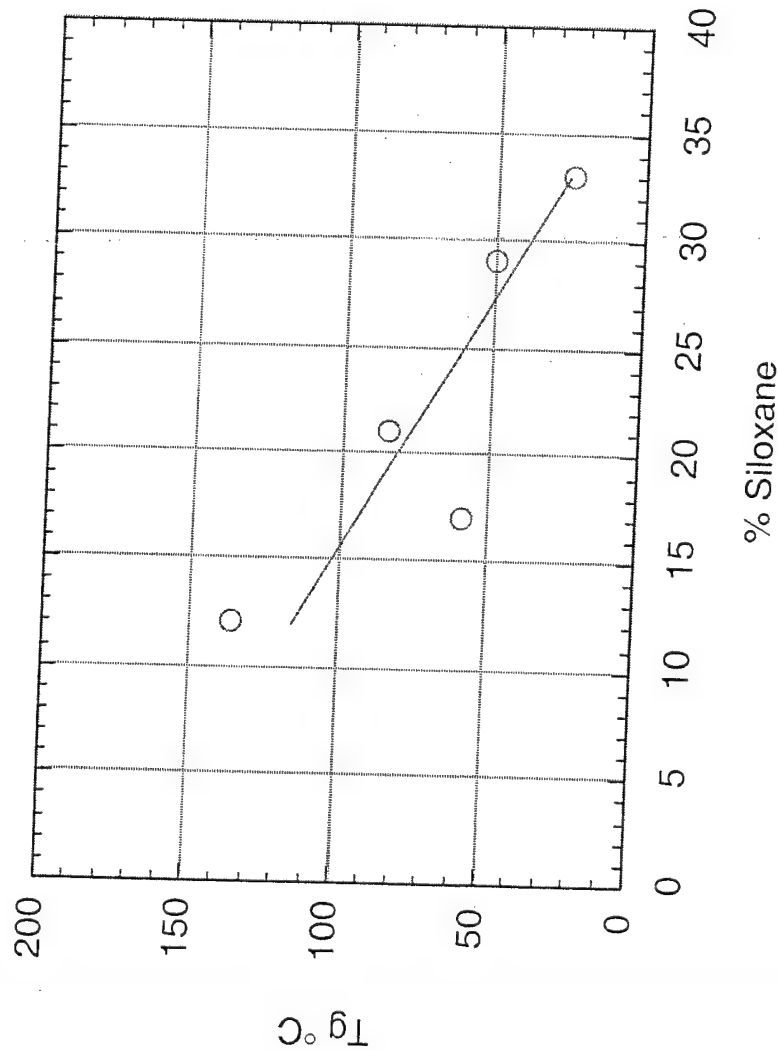
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Tg's For Bead Siloxane Copolymers



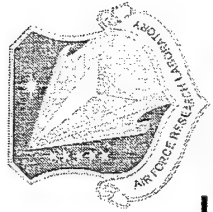
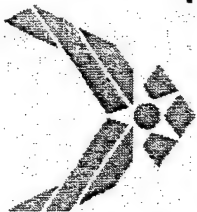
Glass Transition vs % Siloxane



POSS Bead acts as a hard segment

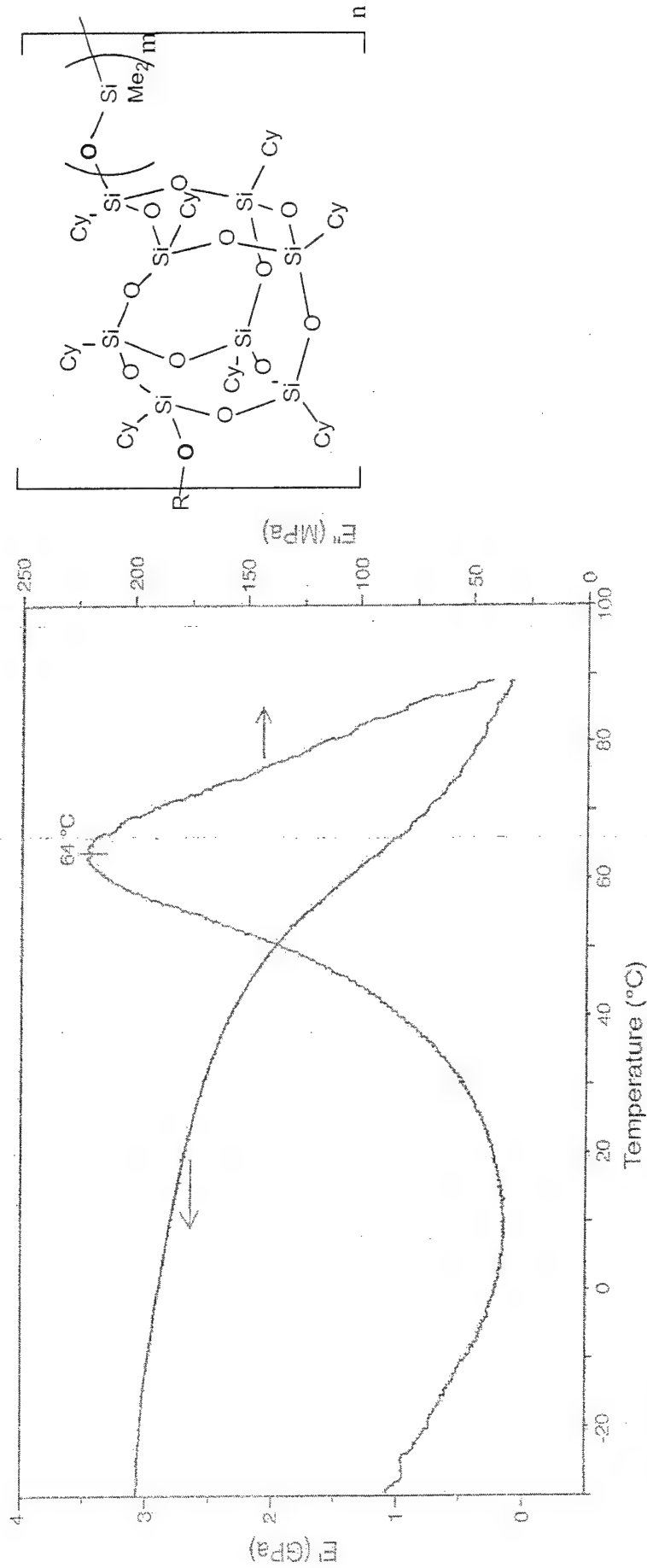
PAS-03-061

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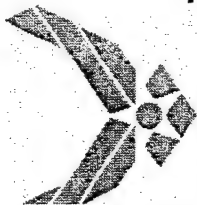
POSS Siloxanes

- Copolymers with low softening temperatures can be molded into bars.
- Dynamic mechanical analysis reveals a T_g (64°C).

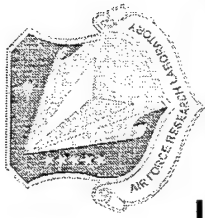


PAS-03-061

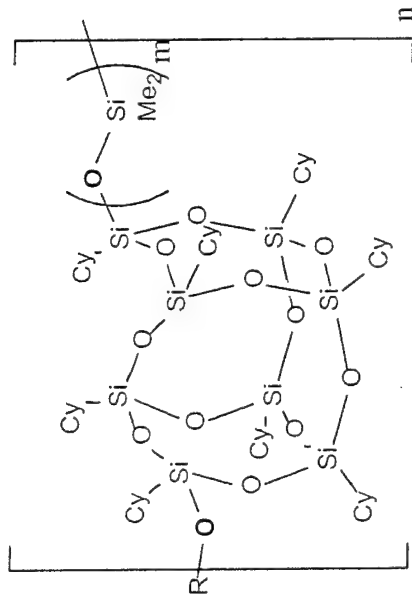
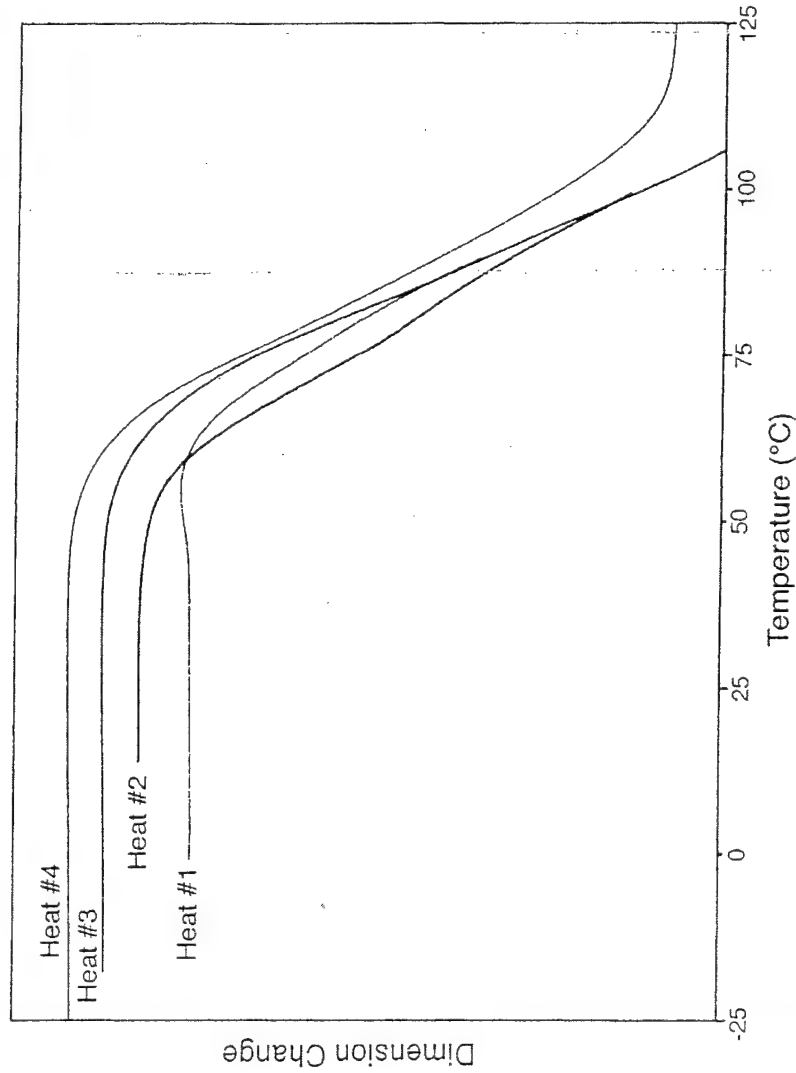
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POSS Siloxanes

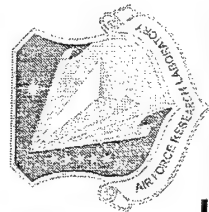
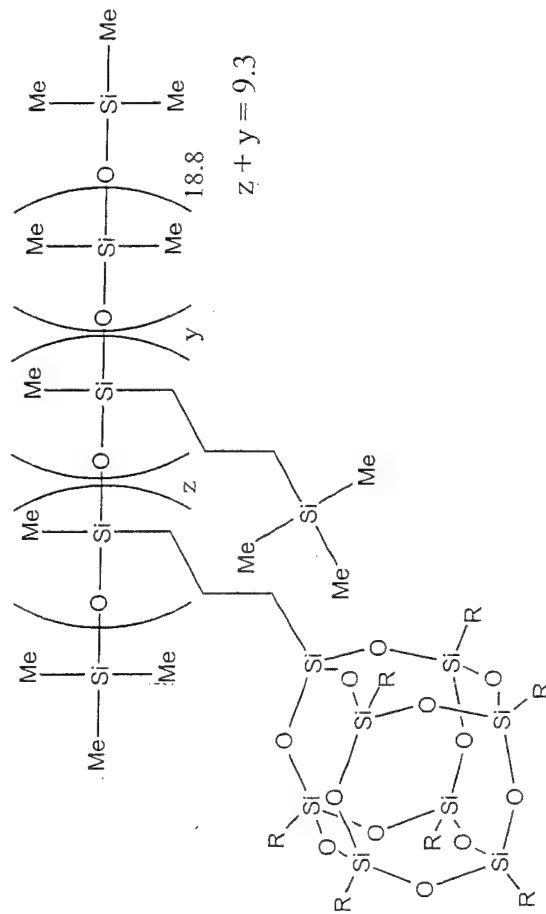


The POSS/Siloxane copolymers with four or more Si-O repeat units in the siloxane segment have softening temperatures well below the decomposition temperatures.

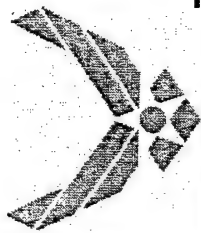


PAS-03-061

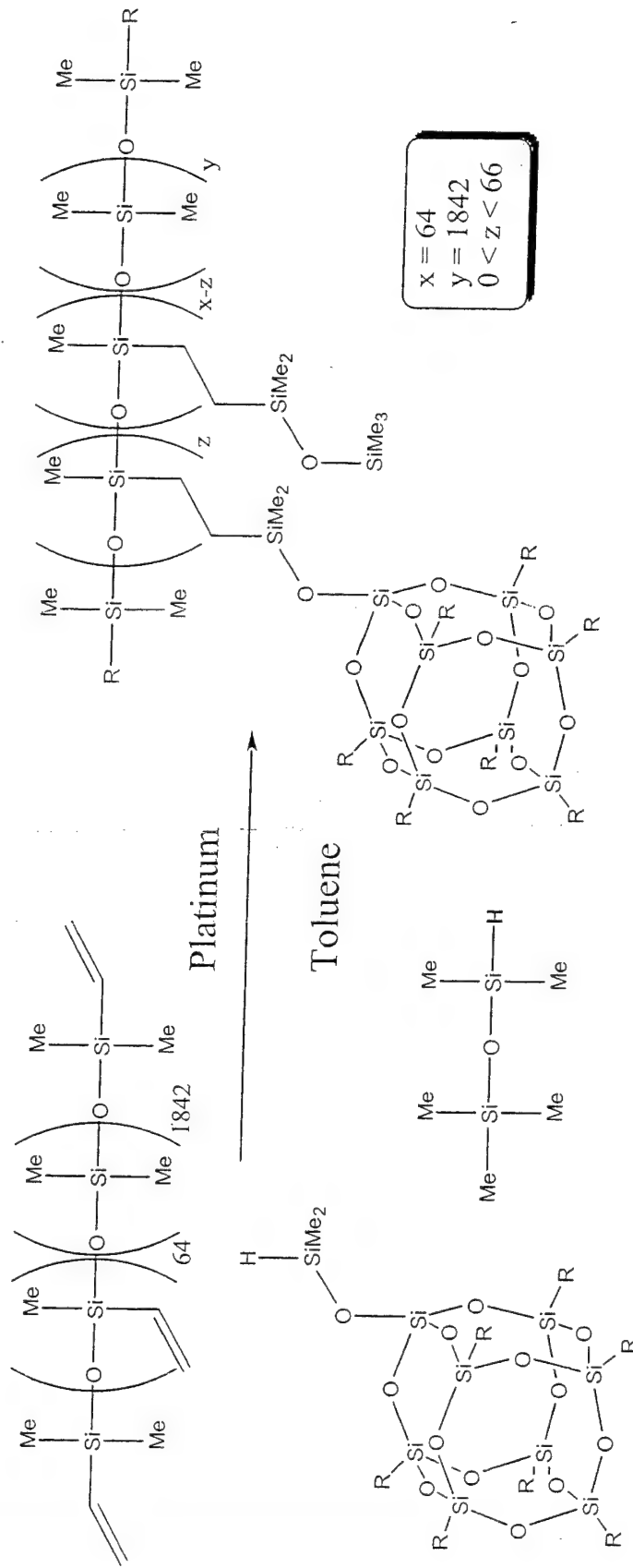
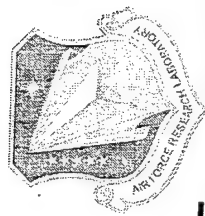
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[illegible]

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Hydrosilation to High MW PDMS



There are about 7 POSS-
macromers per PDMS chain

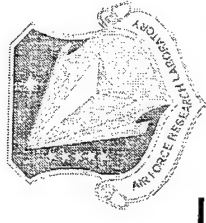
Used 5 weight % POSS

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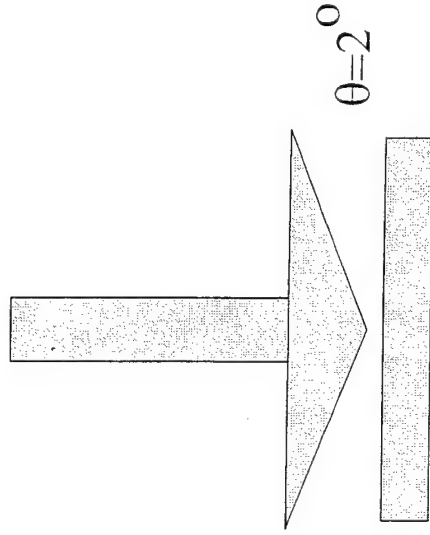
Experimental Setup for Rheology



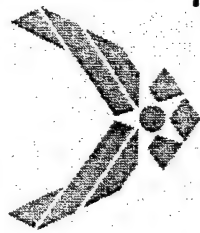
- 25 mm diameter cone-and plate with cone angle of 2° was used.
- The strain amplitude γ_o is 1% and angular frequency ω is 2π per second.
- The temperature is ramped from - 60°C to 70°C with a rate of $2^\circ\text{C}/\text{min}$.

$$\gamma(\omega) = \gamma_o \sin(\omega t)$$

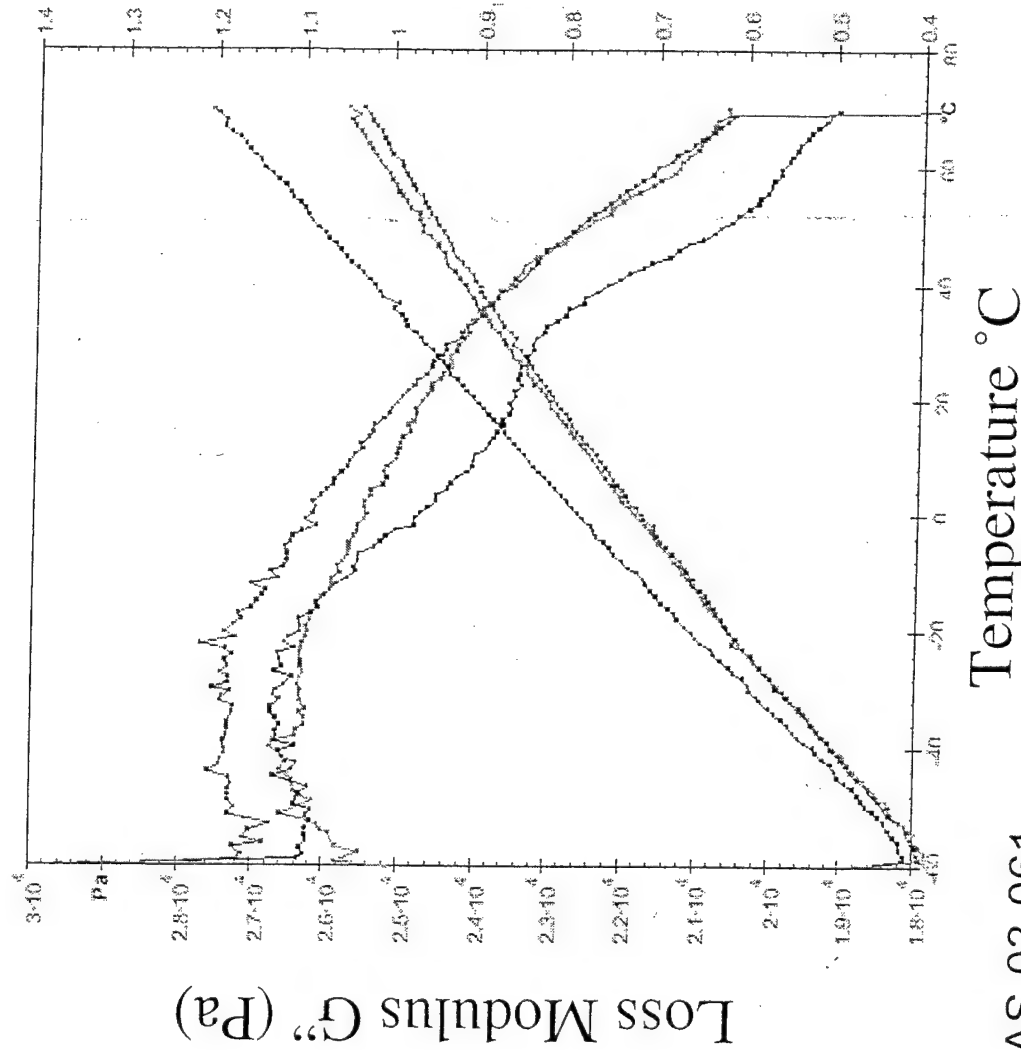
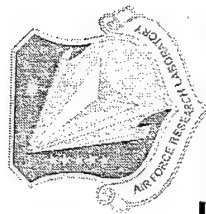
$$\omega = 2\pi (\text{sec}^{-1})$$



The loss modulus G'' and $\tan\delta = G''/G'$ were obtained as a function of temperature.



Comparison of Three T8-POSS Macromers

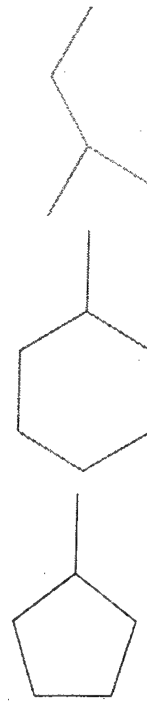
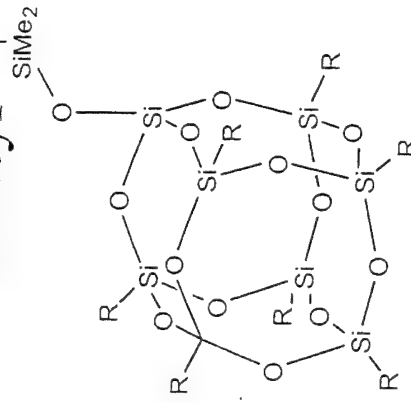


PDMS + 5 wt % POSS

Blue = cyclopentyl

Red = cyclohexyl

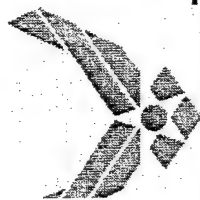
Purple = isobutyl



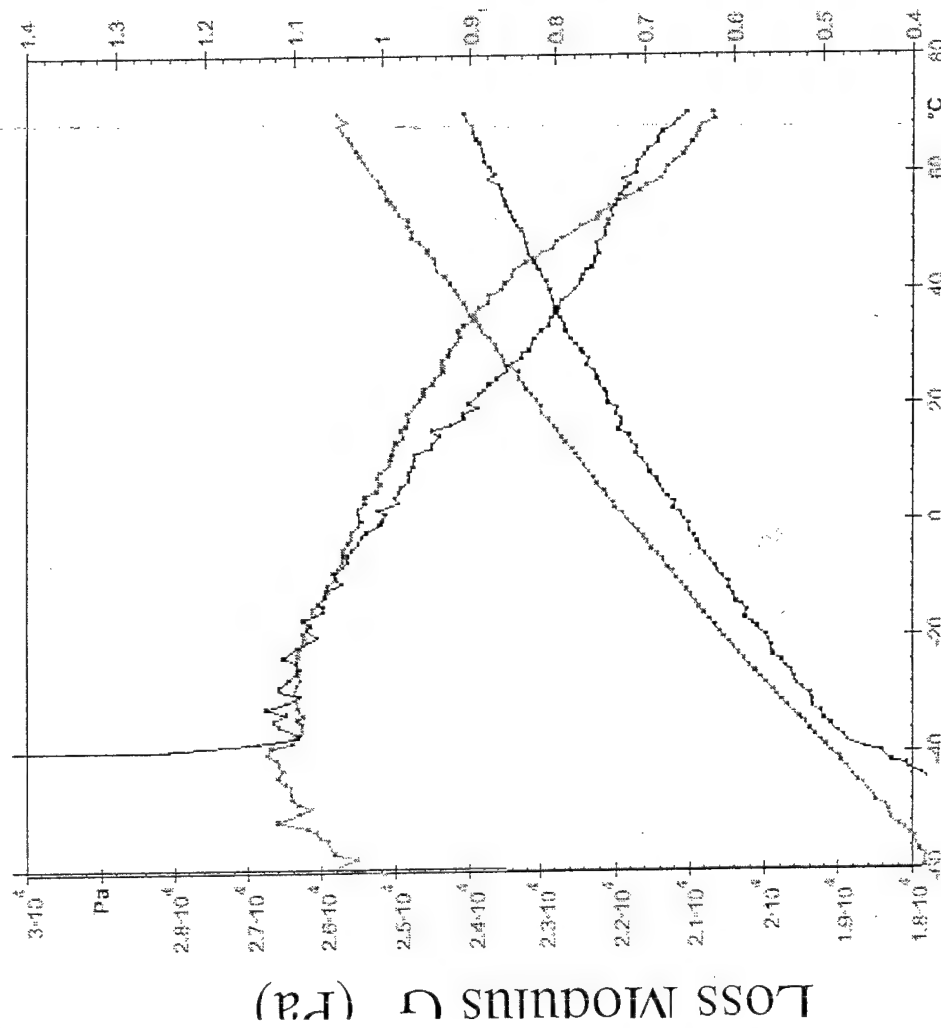
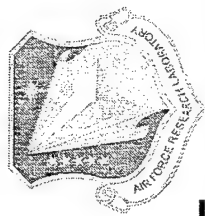
PAS-03-061

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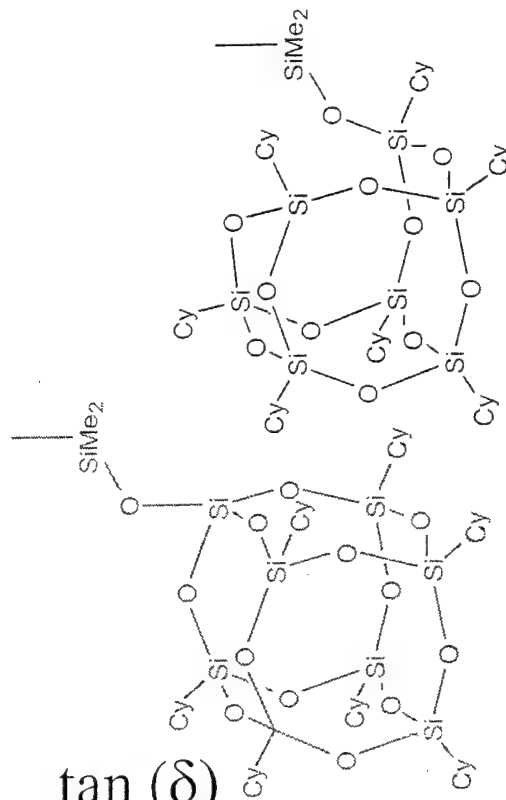
Andre Lee, AFRL



Comparison of Two POSS Polyhedra



$\tan(\delta)$



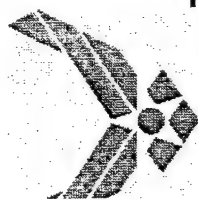
PDMS + 5 wt %
CyclohexylPOSS
Red = T8-POSS
Blue = T7-POSS

Temperature $^{\circ}\text{C}$

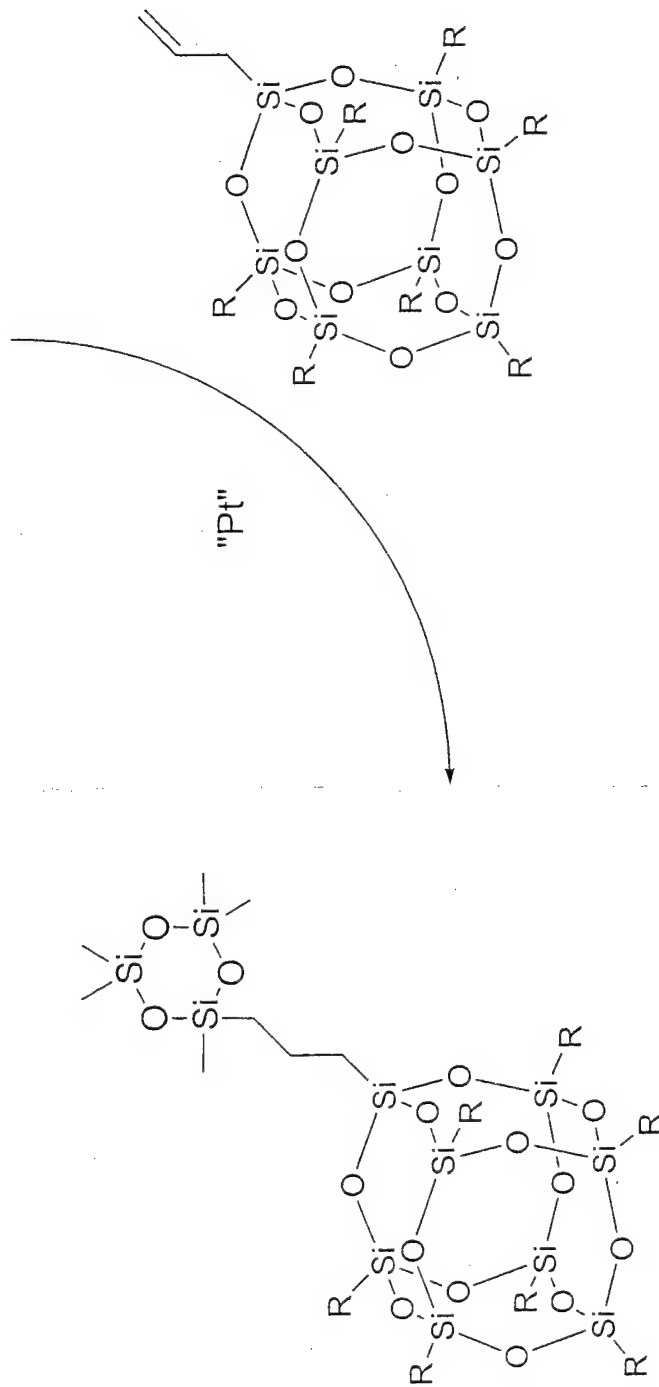
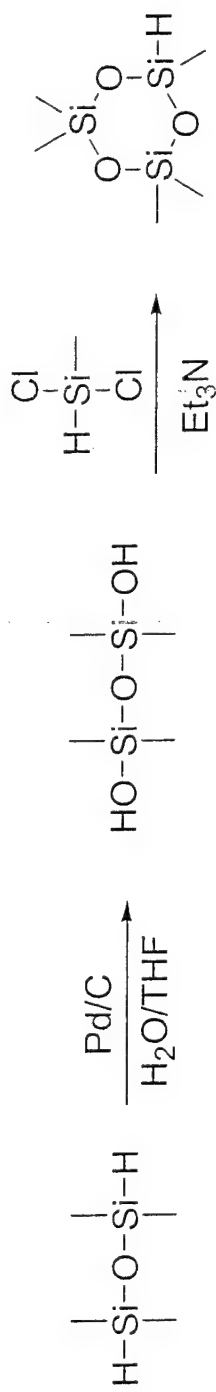
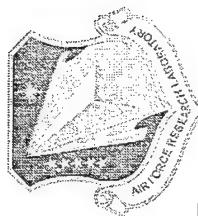
Continue this collaboration with Andre Lee

PAS-03-061

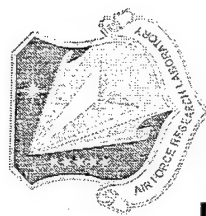
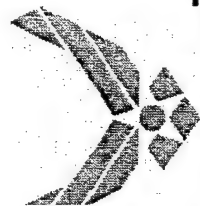
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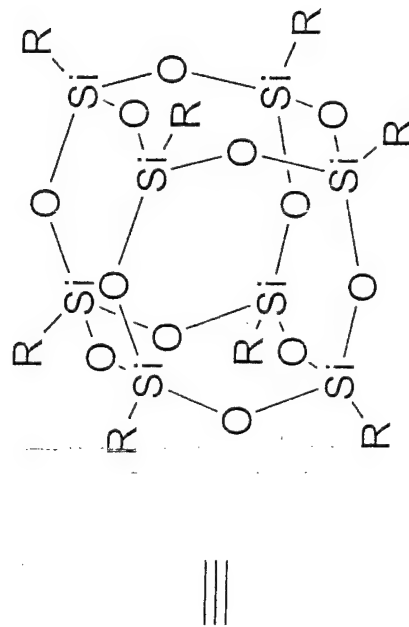
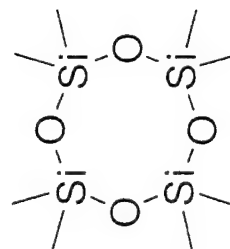
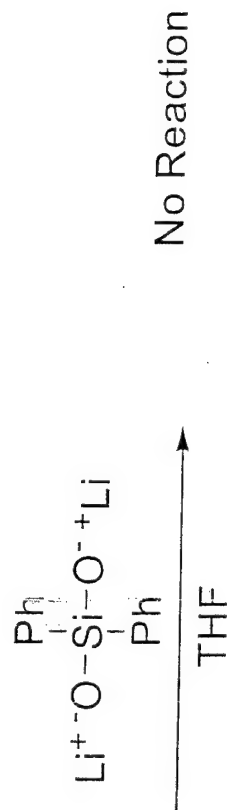
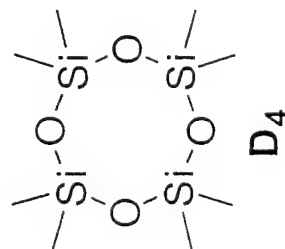
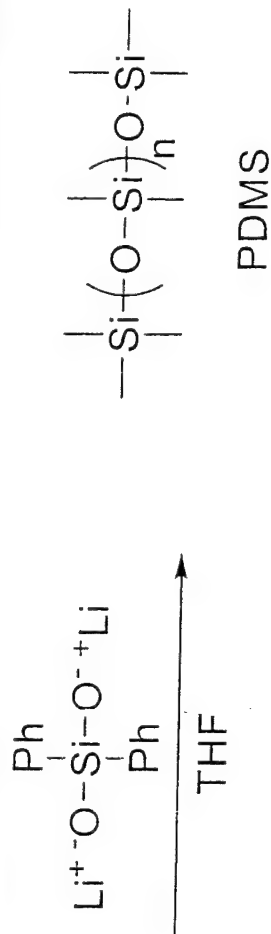
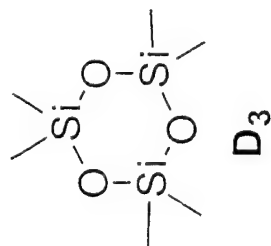
POSS Cyclotrisiloxane

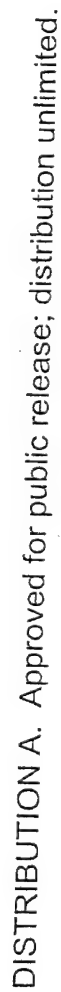
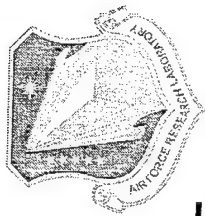


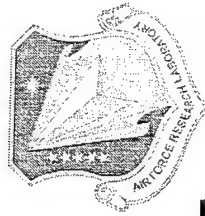
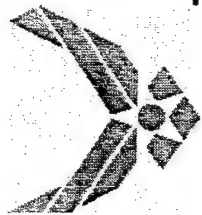
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Reactivity



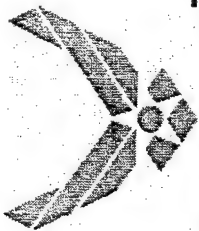




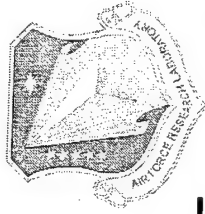
Space-Survivable Materials

PAS-03-061

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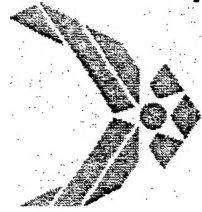


Space Materials

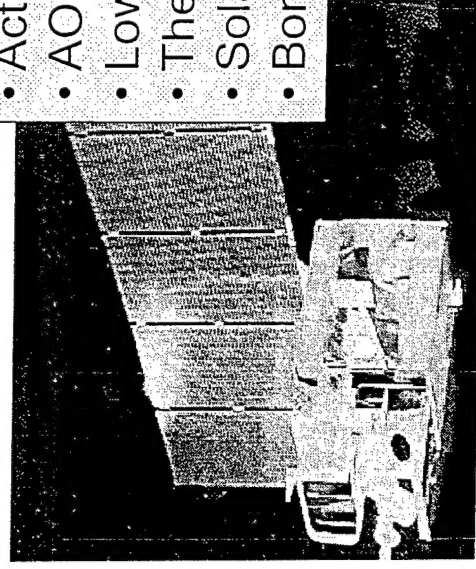
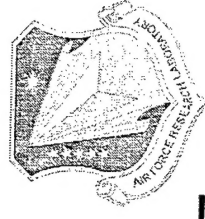


- The International Space Station, the Space Shuttle, and the Hubble Space Telescope are among satellites that operate in Low Earth Orbit (LEO)
- Metallized Teflon FEP is commonly used as outer layer of multi-layer insulation.

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Space-Survivable Polymers



Satellites & Space Systems

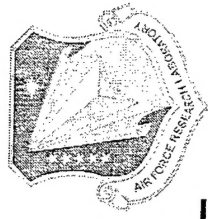
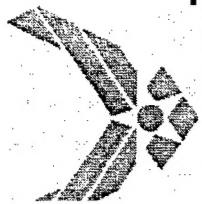
LEO Environment (Altitudes of 200 to 1500 km)

- Atomic Oxygen (AO): $\sim 10^8$ atoms/cm³
- Actual AO flux on spacecraft $\sim 10^{15}$ atoms/cm²•s
- AO Collision energy $\sim 5\text{eV}$ (7.8 km/sec)
- Low-energy and high energy charged particles.
- Thermal cycling -50 to 150°C
- Solar VUV and UV radiation ($\sim 150 - 400$ nm).
- Bond scission and radical formation can lead to embrittlement.

Bond	Dissociation Energy (eV)	λ (nm)	Material
CF ₂ -CF ₂	4.3	290	FEP Teflon®
CF ₂ -F	5.5	230	FEP Teflon®
Si-O	8.3	150	Nanocomposite

Objectives

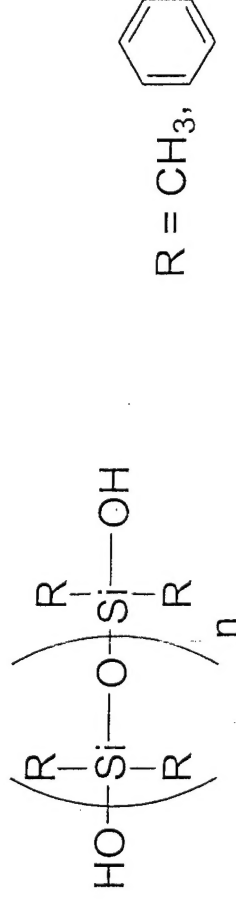
- Increase space-survivability of polymeric materials
- Develop self-passivating layer based on nanocomposite incorporation



Siloxanes

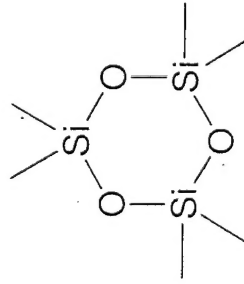
Siloxanes systems exhibit superior resistance to AO

- High Si-O bond strength ~ 8 eV



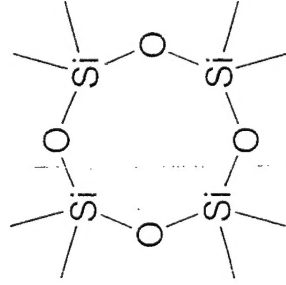
However, pure siloxane systems have disadvantages

- Volatile cyclic species which recondense on optical surfaces



Cyclo(Me₂SiO)₃

(D₃)



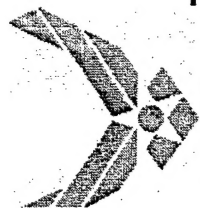
Cyclo(Me₂SiO)₄

(D₄)

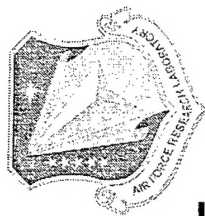
Cyclo(Me₂SiO)₅ (D₅)

Cyclo(Me₂SiO)₆ (D₆)

Cyclo(Me₂SiO)₇ (D₇)



POSS-Siloxane

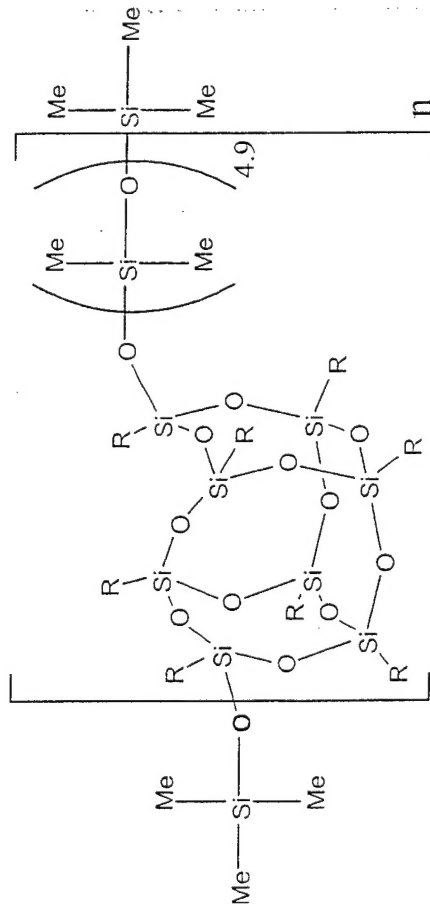


After AO exposure, POSS siloxane had no erosion

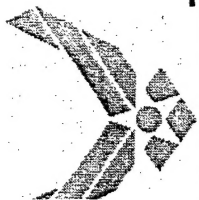
Composition, at %

Sample Treatment	C	O	Si
As entered	65.0	18.5	16.6
2.0 hr	48.4	33.8	17.8
24.6 hr	22.1	49.1	28.8
63.0 hr	16.3	55.7	28.0
4.8 hr air	19.5	52.8	27.7

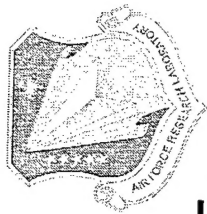
Gonzalez, R. L., Phillips, S. H., Hoflund, G. B., *J. of Spacecraft and Rockets*, Vol 37, No. 4, 2000, pp. 463-467.



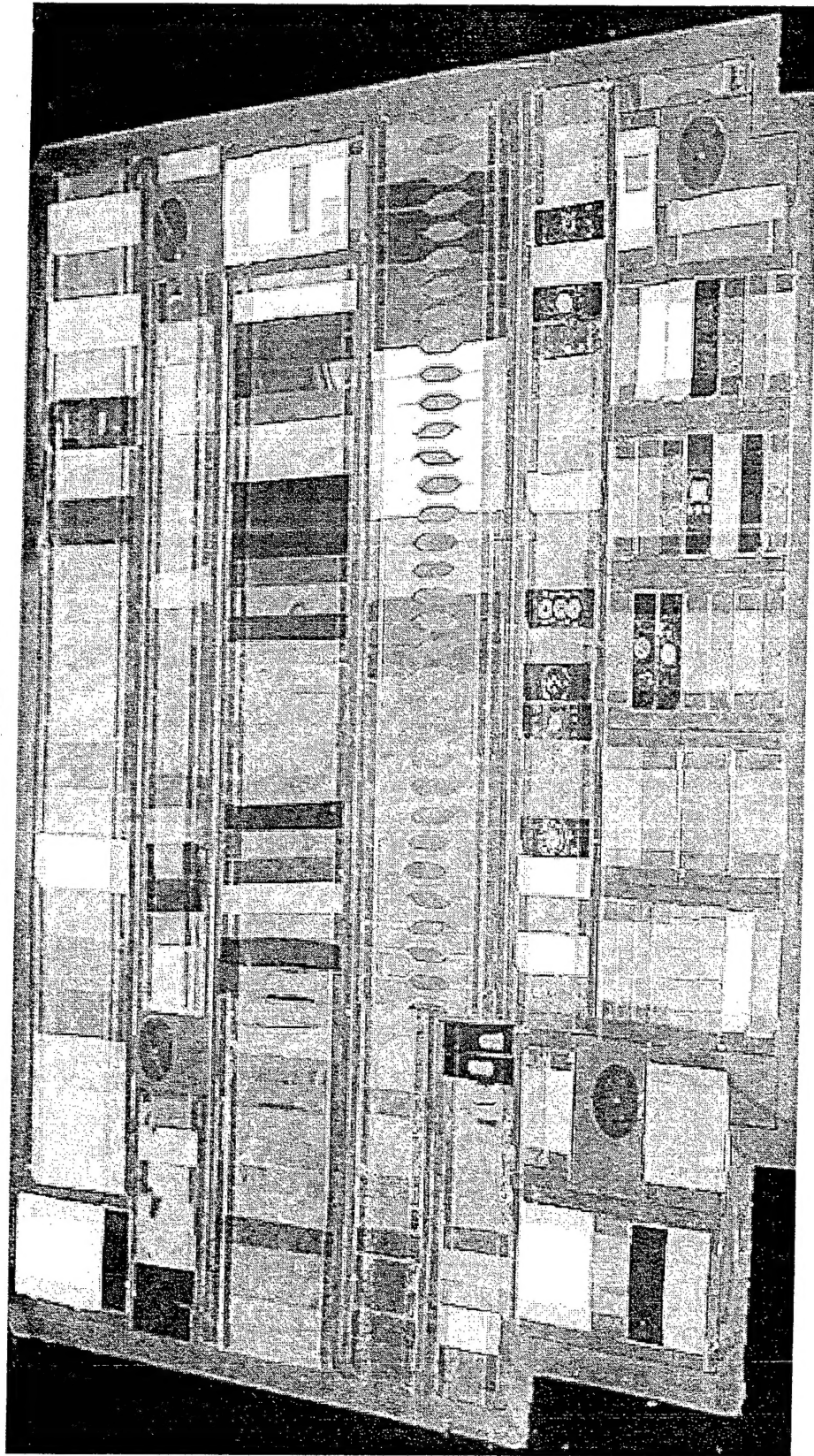
XPS survey spectra obtained from a solvent-cleaned, POSS-PDMS film.



Materials International Space Station Experiment (MISSE)



MISSE 5



Pictures courtesy of NASA

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